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Membrane Processing of Cottage Cheese Whey for Pollution Abatement

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MEMBRANE PROCESSING OF COTTAGE CHEESE WHEY FOR POLLUTION ABATEMENT

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An ultrafiltration reverse osmosis system for the treatment of cottage cheese whey has been successfully demonstrated on both the laboratory and pilot plant scale. A protein concentrate is recovered in the ultrafiltration step and the remaining deproteinized whey is concentrated by reverse osmosis to provide a lactose concentrate. Both concentrates can either be processed further or used directly as liquids. 97% BOD reductions, from 35,000 mg/l to less than 1000 mg/l were achieved. Membrane life was excellent and membrane fluxes were economically high. Total plate counts in the pilot plant produced concentrates were typically below 50,000 org/ml, while coliform counts were nil. Total capital cost for a 300,000 lbs cottage cheese whey/day demonstration plant was estimated at $697 000, with total annual operating costs estimated at $220,000. However, 576,000 lbs of protein and 4,320,000 lbs of lactose will be recovered, and sale of these products is expected to meet capital and operating expenses as well as show a highly favorable return on the investment. (Lowry-Texas)

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Membrane Processing of Cottage Cheese Whey for Pollution Abatement

by

Crowley's Milk Company
Binghamton, New York

for the

OFFICE OF RESEARCH & MONITORING

ENVIRONMENTAL PROTECTION AGENCY

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ABSTRACT

A two-step membrane process has been demonstrated for the treatment of cottage cheese whey. The process produces valuable protein and lactose by-products while reducing the BOD of the whey effluent. The process has been studied in detail in prototype experiments at Abcor, Inc., Cambridge, Mass., and in a 10,000 lbs/day pilot plant at Crowley's Milk Company, Binghamton, New York.

In the two-step process, a protein concentrate is first recovered in an ultrafiltration operation. In the second step, ultrafiltration permeate (de-proteinized whey) is concentrated by reverse osmosis to provide a lactose concentrate. The protein concentrate can be further concentrated and/or dried; and the lactose concentrate can be further concentrated and lactose recovered by crystallization, or otherwise processed. Alternatively, the membrane concentrates can be used directly as fluid products.

Operation of the pilot plant was successful and almost troublefree. BOD reduction of the raw whey was about 97%, from an initial value of about 35,000 mg/l to less than 1,000 mg/l. Membrane life was excellent, and membrane fluxes were economically high.

The pilot plant produced protein and lactose products with low total plate counts, and nil coliform counts. Using the cleaning procedure developed in the prototype program, total plate counts were typically below 50,000 org/ml.

Projected capital cost for a 300,000 lbs cottage cheese whey/day demonstration plant is $697,000. This includes both the ultrafiltration and reverse osmosis sections of the plant, tanks, and a building to house the plant. Projected operating costs are $220,000 per year. Projected income from utilization of the protein and lactose concentrates makes the process profitable and results in an attractive return on investment.

This report was submitted in fulfillment of the requirements of Grant No. 12060 DXF given by the Environmental Protectio. Agency to Crowley's Milk Company.

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SECTION I

CONCLUSIONS

1. Membrane processes offer an economically attractive solution for waste treatment of cottage cheese whey. Pilot operation has shown that a two-step process sucessfully produces protein concentrates through whey fractionation by ultrafiltration and lactose concentrates through reverse osmosis.

2. The BOD of raw cottage cheese whey can be reduced from approximately 35,000 mg/l to less than 1,000 mg/l by membrane processing.

3. Protein concentrates containing up to 20% protein (80% protein on dry solids basis) can be generated in a single step ultrafiltration.

4. Lactose concentrates containing 20% lactose (75% lactose on a dry solids basis) can be generated by concentration through reverse osmosis of the ultrafiltration permeate.

5. Whole cottage cheese whey can be concentrated to about 25% solids by reverse osmosis without prior deproteinization (by ultrafiltration or otherwise).

6. Products produced from the two-step membrane process, protein and lactose concentrates, have low total plate counts and nil coliform counts. Products produced in the pilot plant operation met standards for Grade A milk.

7. Concentration of whole whey by reverse osmosis may be more difficult than concentration of deproteinized whey because clean-up and sanitizing of the membrane equipment is complicated by the presence of casein fines and protein.

8. Installation and operation of a 300,000 lbs/day plant are expected to yield a high return on investment before taxes, corresponding to a rapid plant pay-out. This conclusion incorporates both capital and operating costs, as well as information proprietary to Crowley's Milk Company regarding the utilization and value of protein and lactose products.

9. Plant capital cost for a 300,000 lbs whey/day installation is projected to be $697,000; and annual operating cost, $220,000.

SECTION II

RECOMMENDATION

Crowley's Milk Company recommends the installation and operation of a 300,000 lbs cottage cheese whey/day membrane processing facility at the LeFargeville, N.Y. plant. This unit is to be constructed in Phase II of the program.

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SECTION III

INTRODUCTION

The status of the pollution problem resulting from the disposal of acid cheese whey is such that new methods must be used to deal with it effectively. This section of the report presents a summary of the size and nature of the waste problem and provides a general description of the membrane separation process under demonstration.

BACKGROUND AND NEED FOR PROJECT

The manufacture of cheese from either whole or skim milk produces, in addition to the cheese itself, a greenish-yellow fluid known as whey. Whole milk is used to produce natural and processed cheeses such as cheddar, and the resulting fluid by-product is called sweet whey, with a pH in the range of 5 to 7. Skim milk is the starting material for cottage cheese and gives a fluid by-product called acid whey, with a pH in the range of 4 to 5. The lower pH is a result of the acid developed during or employed for coagulation.

Each pound of cheese produced results in five to ten pounds of raw fluid whey. The high organic content of whey leads to a severe disposal problem. Over 70% of the nutrients from skim milk show up in acid whey, including proteins and lactose. These materials, if properly recovered, could provide useful products. Table 1 shows typical compositions of whey and dried whey solids.

Production of Cottage Cheese Whey:

Some statistical information on the production of cottage cheese (acid) whey is summarized here to provide a basis for identifying the needs for acid whey treatment. In categorizing industries in the United States, cottage cheese is considered to be part of fluid milk rather than cheese production. Thus statistics on acid whey are somewhat more difficult to obtain than those for cheddar and other sweet wheys. However, the general trends in the fluid milk industry are the same as those for the natural cheese industry, i.e., the industry is growing, but the number of plants is decreasing. These trends are shown in Figure 1.[1] The trend towards exceptionally large regional plants with distribution over wide areas is particularly notable.

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TABLE 1

AVERAGE COMPOSITION OF WHEY PRODUCTS

Product	Water	Nitrogenous Matter	Fat	Lactose	Acid	Ash
Raw Cheese Whey	93-94%	0.7-0.9%	0.05-0.6%	4.5-5.0%	0.2-0.6%	0.5-0.6%
Condensed Whey	50-60%	7-8%	1-2%	28%	1-3%	5-6%
Dried Whey	2-6%	12-14%	0.3-5.0%	65-70%	2-8%	8-12%

By 1969, overall production of cottage cheese curd and creamed cottage cheese had risen to an adjusted value of 966 million pounds. The acid whey output accompanying this value was set at 4.83 billion pounds, based on 5 pounds whey per pound of cottage cheese. A present output of at least 5 billion pounds of acid whey per year is considered to be a conservative value, based on the current rate of growth of cottage cheese production and on the fact that statistics for other related acid cheese wheys (bakers, farmers, cream) are not included.

The Cottage Cheese Whey Disposal Problem:

The organic nutrients of whey, which go unused, place a costly burden on sewage systems and waterways. The biological oxygen demand (BOD) of whey ranges from 32,000 to 60,000 ppm. Most of this BOD is due to the lactose. Specific BOD values for cottage cheese wheys are between 30,000 and 45,000 mg/l, depending primarily on the specific cheese making process used.

Every 1000 gallons per day of raw whey discharged into a sewage treatment plant can impose a load equal to that from 1800 people. This is partially passed into streams in most cases because BOD removal is not complete. Every 1000 gallons of raw whey discharged into a stream requires for its oxidation the dissolved oxygen in over 4,500,000 gallons of unpolluted water.

Recent statistics give BOD's of about 0.2 pounds per pound of cottage cheese curd. Combining this with the production statistics given above, a total BOD removal of over 200 million pounds is required for complete waste treatment. Considering that at the very best only half of the cottage cheese whey produced is currently put to good purpose and that curd wash water can contain 3% solids, the disposal problem is severe.

PROPOSED MEMBRANE PROCESS

A two-step membrane separation process has been developed for the treatment of cottage cheese whey. Although the demonstration has been for acid whey treatment, the technology is applicable to sweet wheys. In this process, whey is simultaneously fractionated and concentrated to give protein and lactose by-products. The final effluent has a low biological oxygen demand (BOD) and is expected to be suitable for reuse within the cottage cheese plant. One application, for example, is in curd washing. If the effluent is discharged, a final treatment for residual BOD removal may be required, depending on local and state regulations.

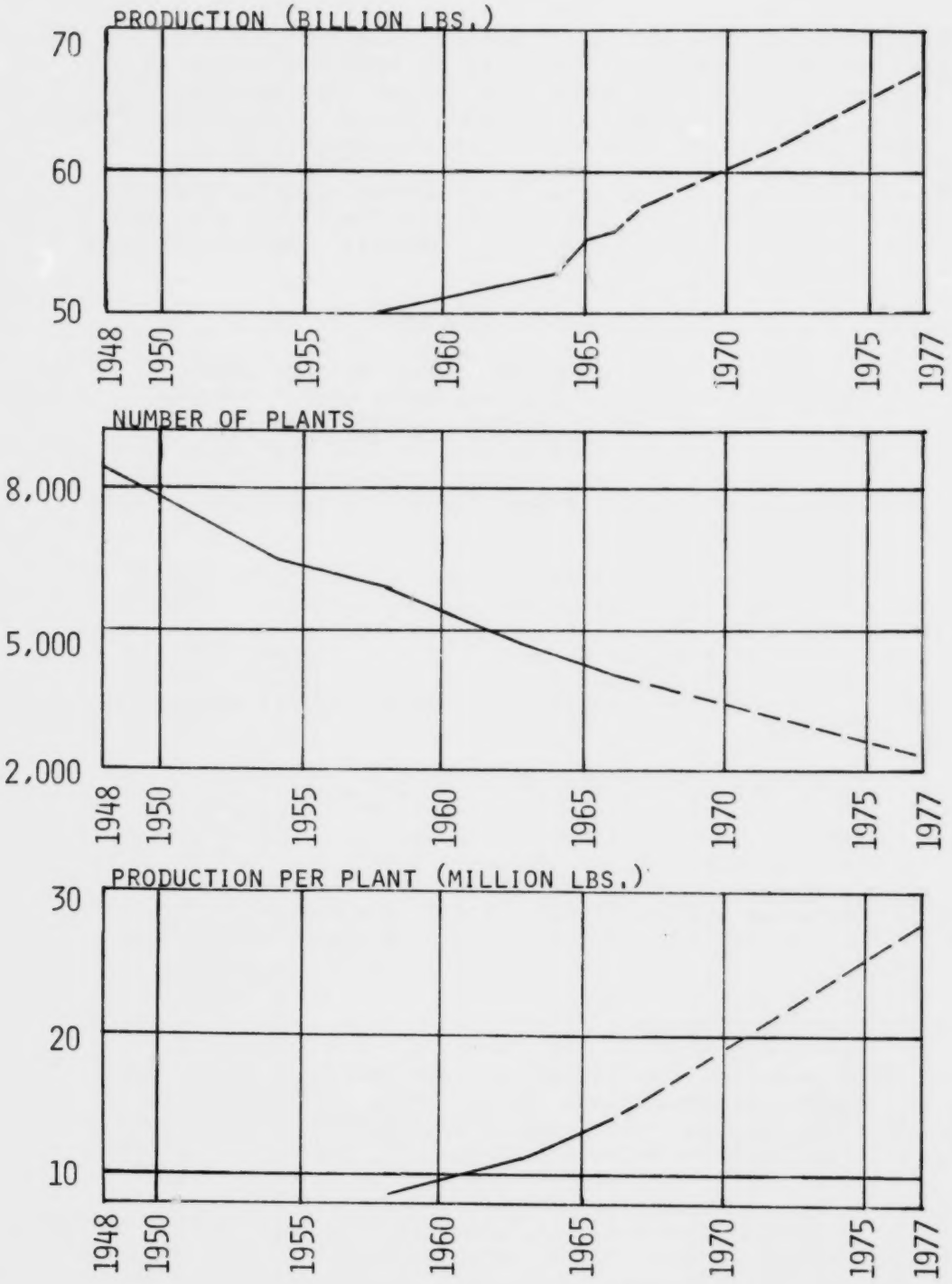

FIGURE 1: TRENDS IN FLUID MILK PRODUCTION (1)

The two-step whey treatment process is based on the application of ultrafiltration (UF) and reverse osmosis (RO). Reverse osmosis has been extensively studied during the past ten years under funding from the Office of Saline Water, the bulk of this work focusing on desalination of brackish and sea waters. Additional publicity has been given to its application to whey concentration by the United States Department of Agriculture.(3,4,5) Ultrafiltration is a variation of this membrane separation technique.

Figure 2 shows the basic concept involved. A semi-permeable membrane separates water and a solution. In the absence of a hydrostatic pressure differential, water will permeate the membrane so as to dilute the solution. A counter pressure can be applied to the solution side to reverse water transport. The amount of pressure required to achieve a static equilibrium is termed the osmotic pressure. Reverse osmosis is simply the application of a pressure greater than the osmotic pressure which drives water from the solution side of the membrane to the water side, and permits concentration of the solution.

The membrane plays the most critical role in the process. By varying the properties of the membrane, one can control the retention or passage of selected solutes. When the solute molecules are large, for example whey proteins, the osmotic pressure is quite low and a membrane with relatively large pores can be used at low operating pressures: e.g., 10 to 100 psi. This is termed ultrafiltration. Referring to Figure 3, the membrane may be called "loose"; that is, lower molecular weight solutes will pass through the membrane and will not be retained in the concentrate.

In reverse osmosis, solutions of small molecules with moderate to high osmotic pressures are retained, and the required driving force is considerably higher, ranging from several-hundred psi to over one-thousand psi. Higher operating pressures are required because of the substantial osmotic pressure of salt and sugar solutions, and also because of the greater resistance to water transport of RO membranes. Again referring to Figure 3, a "tight" membrane is employed.

Figure 4 shows a simplified flow sheet for the two-step whey treatment process. Cottage cheese whey, with or without filtration for fines removal, is introduced into a low pressure UF unit (step 1). In this operation, whey is concentrated 10 to 30-fold by volume. Ultrafiltration membranes are used which retain only the whey proteins. Thus, it is

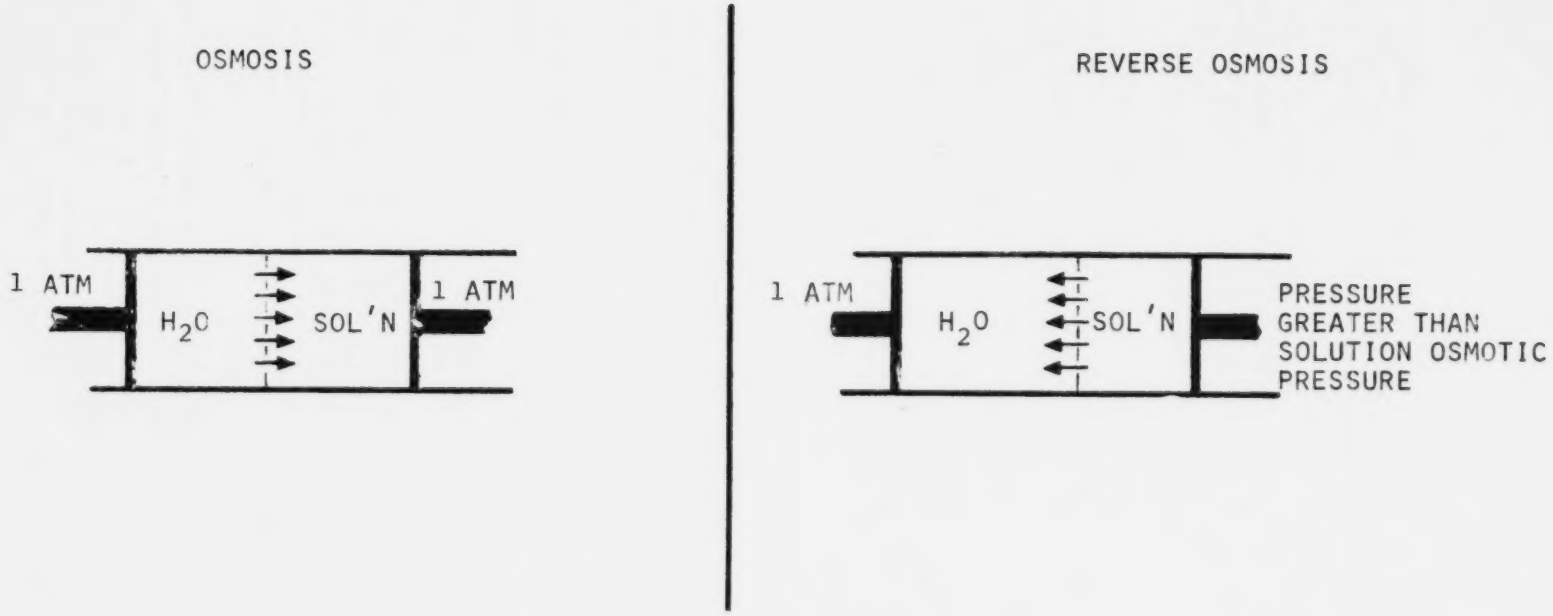

FIGURE 2

OSMOSIS AND REVERSE OSMOSIS

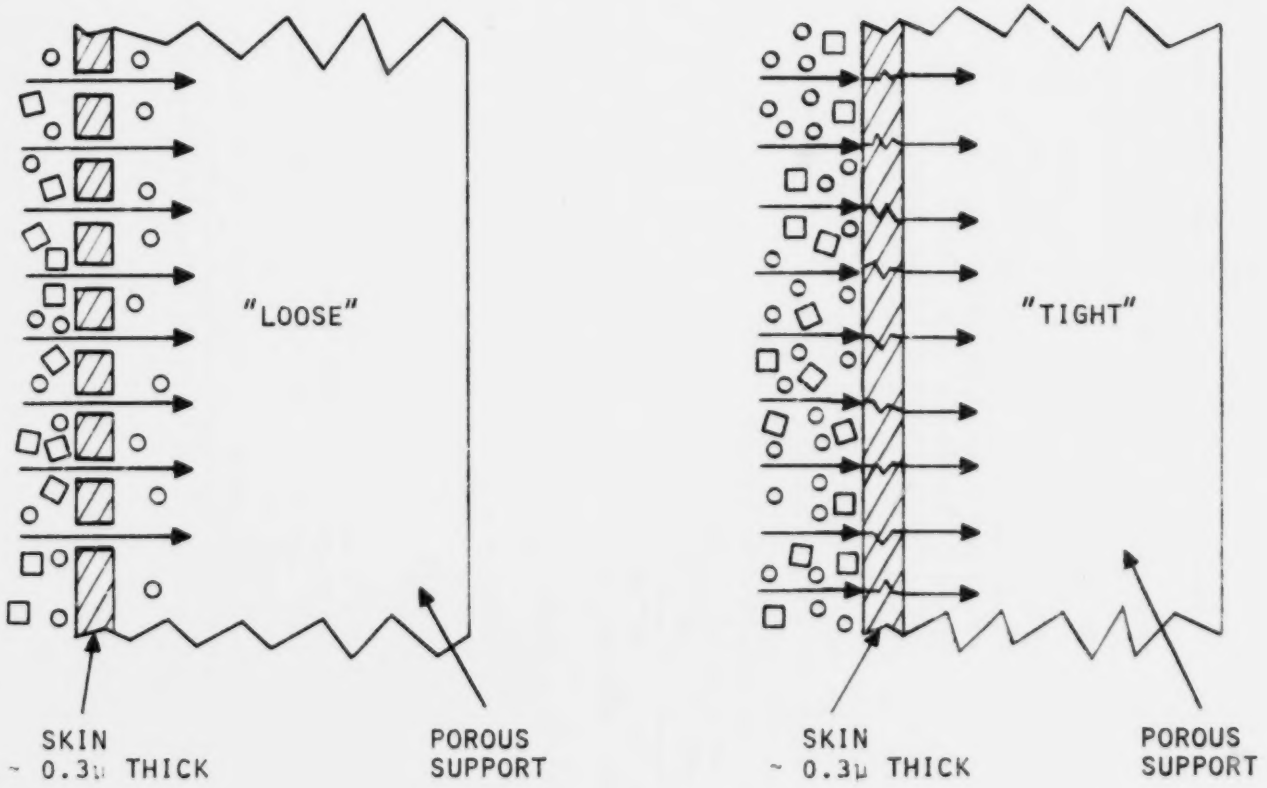

FIGURE 3

MECHANISM OF MEMBRANE SEPARATION

possible to obtain a protein concentrate with a higher proportion of proteins in the dissolved solids, since lactose, non-protein nitrogen, lactic acid, and minerals pass through the membrane. Operation is typically in the pressure range of 10 to 100 psi, and at temperatures of 60 to 130°F. As will be demonstrated in the data presented below, the protein content of raw whey can be increased from an initial value of about 0.6% up to levels approaching 20% in this step. Simultaneously, the proportion of protein in the whey solids can be increased from approximately 10% to 80% in a one-step concentration.

The permeate (ultrafiltrate) from the UF unit is introduced into a second membrane step. In an RO operation, this stream is concentrated from approximately 6% solids to 20-25% solids. Typical operation would be in the pressure range, 500 to 1500 psi, and at a temperature of 60 to 100°F. The membrane in the RO section is chosen so as to retain as great a proportion of the organic solutes as possible, resulting in the permeate having a low BOD. The final effluent from the RO section can either be reused within the dairy or cheese plant or discharged, with or without treatment for residual BOD removal. Where pollution control regulations are not overly stringent, a moderate salt rejection membrane can be used in the RO section to permit partial desalting and lactic acid removal from the lactose concentrate. In general, from the point of view of pollution control, this option will probably not be exercised.

The protein concentrate can either be used directly by incorporation into food products, or it can be dried. Drying may be preceded by concentration by vacuum evaporation. The lactose concentrate can be further concentrated by evaporation and the lactose can be recovered in a simple crystallization operation.

Another process alternative, which has not been explored in depth in this program, is the direct concentration of whole whey by RO. This approach has been examined by others (2,3,4), and appears to be attractive for cheese producers with small volumes of whey. Concentration on site would precede transportation to, and processing at, a central facility. However, a satisfactory cleaning program needs to be defined prior to wide use.

In the immediately following sections, data for the operation of the UF and RO sections will be presented. The data presented first were obtained with prototype systems operated at Abcor, Inc., Cambridge, Massachusetts. The latter data are from operation with a 10,000-to-20,000-lbs whey/day pilot plant operated at Crowley's Milk Co., Binghamton, N.Y.

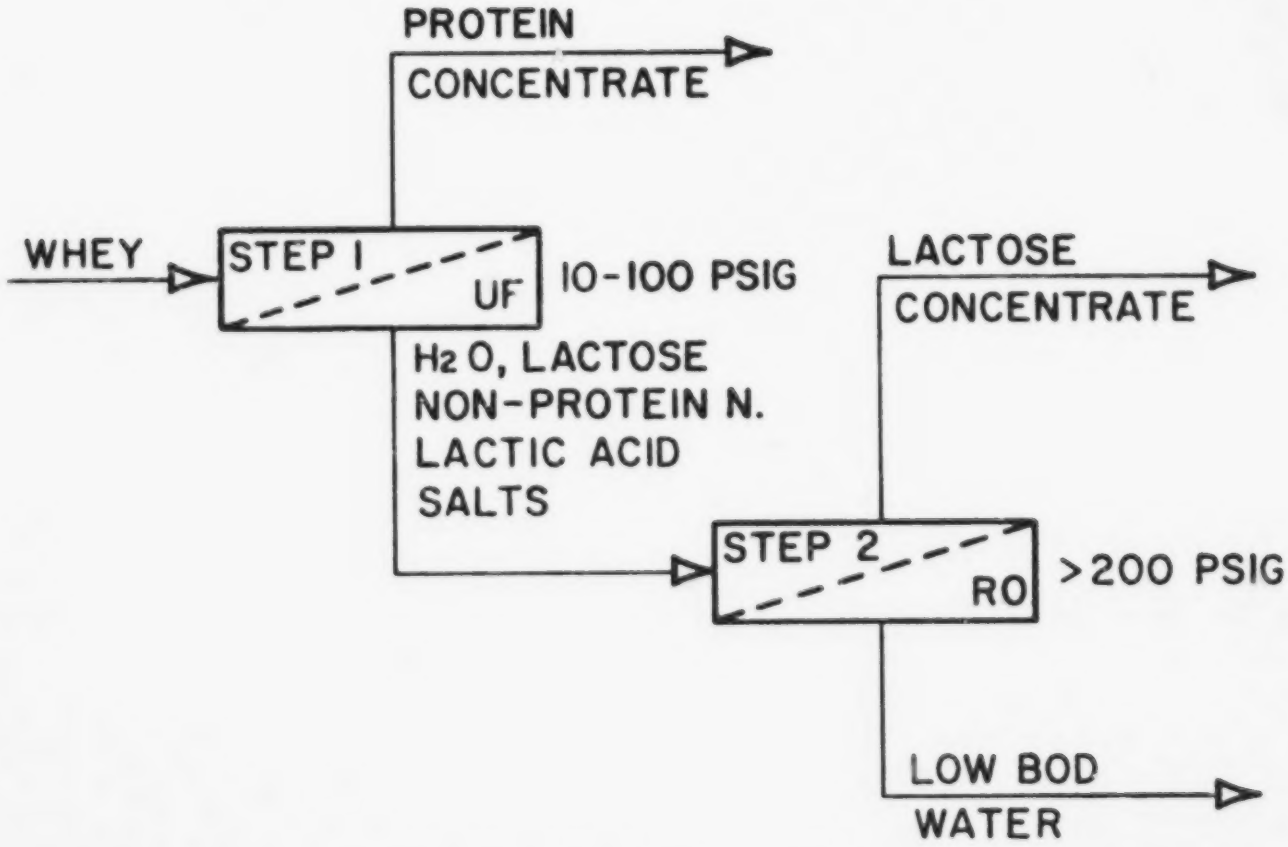

FIGURE 4 -MEMBRANE PROCESS FOR WHEY TREATMENT--FLOW SCHEMATIC

SECTION IV

PROTOTYPE EXPERIMENTS

Extensive prototype experiments were performed prior to the design, installation and operation of the pilot plant. These were conducted at Abcor, Inc. from November, 1969 through June, 1970, under subcontract to Crowley's Milk Co. In the course of these experiments, a variety of membranes and membrane equipment were examined for both UF and RO. Significant experimental results are presented and discussed in this section.

ULTRAFILTRATION

Experimental Procedure

Apparatus and Procedure. Two systems were used to generate the data which follow. The first, containing 13.2 ft^2 of Abcor HFA-180 tubular membranes, was used to batchwise concentrate cottage cheese whey. A flow schematic is shown in Figure 5. Cheese whey was charged to the feed tank and heated to the desired operating temperature by passing hot water (150°F) through a coil installed in the tank. Temperature was maintained with a temperature switch, installed in the tank, which actuated a solenoid valve controlling hot water flow. With this system it was possible to control the temperature of the whey to within 4°F.

Whey was circulated through the tubular membranes with a 2 HP centrifugal pump. The system circulation rate was measured with a rotameter and controlled with two globe valves. These valves were also effective for regulating the system operating pressure. Membrane system inlet and outlet pressures were measured with diaphragm-protected gauges. Fractional water removal per pass through the membranes was very small, typically less than 0.001, and the "conversion per pass" was negligible. Correspondingly, the system contents were well-mixed. Permeate was withdrawn continuously, while the concentrated feed was recovered only at the end of the test, by draining the tank, membranes and piping. This is termed a "batch concentration."

A similar system was used for "differential" studies. In this prototype system permeate was returned continuously to the feed tank in order to maintain a time-invariant feed composition and concentration.

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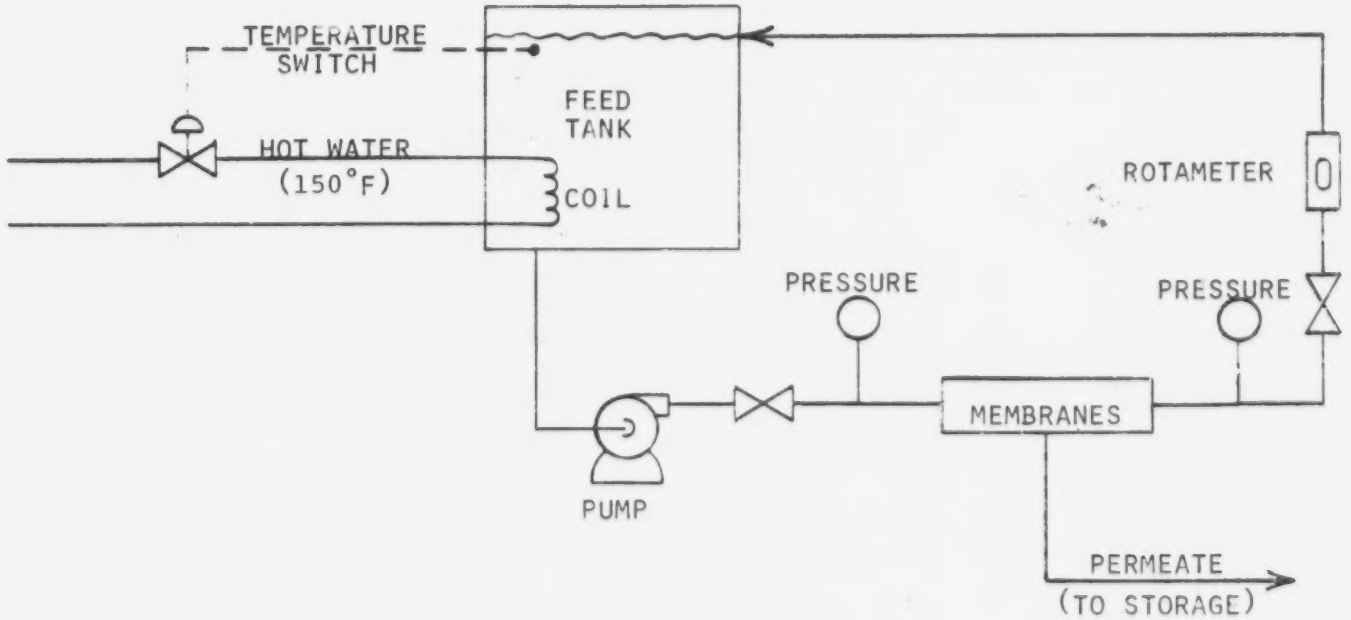


FIGURE 5

TEST SYSTEM FLOW SCHEMATIC

Batch concentration experiments were employed to determine the dependence of membrane flux and membrane retention on feed concentration. Tests of this nature were generally carried out at a fixed temperature, feed circulation rate and pressure. Differential experiments were performed to examine the effects of feed circulation rate, pressure, temperature and time on membrane performance.

Feed Whey. Cottage cheese whey was obtained from two sources. For two experiments whey was shipped from Crowley's Milk Co., Arkport, New York. Unfortunately the delivery time of the whey was sufficiently long such that spoilage of the whey was a problem. For this reason whey for the other experiments was purchase from the H.P. Hood & Sons Co. The whey was shipped from Newport, Maine,immediately after collection and was processed within 24 hours of shipment.

Typical properties of the whey are listed below.

pH	4.5
total acidity (expressed as lactic acid)	0.5-0.65%
lactose	4.5-5%
protein (Kjeldahl)	0.7-0.8%
fat (Roese-Gottlieb)	0.02-.045%
total solids (Mojonnier)	6.2-6.6%
total plate count (when shipped)	500-5,000 org/ml

The cottage cheese whey was processed in the initial experiments without filtration. For later experiments the whey was filtered with a 5μ filter prior to introduction into the membrane system. Filtration for fines removal appeared to have no effect on membrane performance, but may be required when a protein concentrate free of casein fines is desired.

Analytical Procedures. Several analyses were used in the course of the prototype tests. These are listed in Table 2, where references for the procedures and their sensitivities are indicated. Analyses performed at

Abcor, Inc., included COD, Lowry protein, lactose, pH, total acidity and total solids. Analyses for Kjeldahl protein (nitrogen x 6.3) and BOD were carried out at Metcalf & Eddy, Inc., in Boston, Mass. The gel permeation chromatography assays, and the viable microorganism counts were performed at the Department of Nutrition and Food Science at M.I.T., by Professors Stephen R. Tannenbaum and Anthony J. Sinskey, respectively.

For the UF experiments, key factors included the membrane protein retention and the obtainable level of protein concentration in the concentrate. Protein contents presented in this section are Lowry "protein", which was shown to be equivalent to Kjeldahl "protein", based on analysis of identical samples at Abcor and Metcalf and Eddy.

The lactose analysis was also a colorimetric analysis. Standard curves were generated for lactose samples with and without the addition of β-lactoglobulin. The curve without β-lactoglobulin was used to analyze for permeate samples from UF experiments (as well as for the RO tests). The curves with added β-lactoglobulin (at different concentration levels) were used for the analysis of whey and whey concentrate samples.

Abcor's COD analysis was correlated with BOD_5 analyses. A series of samples were frozen and sent to Metcalf and Eddy for analysis for BOD_5. The same samples were analyzed at Abcor. The correlation between BOD_5 and COD was found to be,approximately, BOD=0.78 COD.

RESULTS

Membrane Retention Properties. The retention properties of the ultrafiltration membranes have been examined in detail. Retention is defined by Equation (1):

$$R(\%) = \frac{C_F - C_P}{C_F} \times 100 \qquad (1)$$

where C_F is the feed protein concentration, and C_P is the permeate protein concentration at the corresponding feed concentration. This definition of retention is to be contrasted with yield. Yield is defined as the fraction of protein in the feed whey contained in the whey concentrate. This is given by Equation (2):

TABLE 2: ANALYTICAL PROCEDURES

ANALYSIS FOR:	PROCEDURE:	SENSITIVITY:
COD	Std. Methods for the Exam. of Water, Sewage, and Ind. Wastes, Amer. Pub. Health Assoc., 12th ed., 1965	0.05%
BOD_5	(Metcalf & Eddy)	0.1%
Protein (Lowry)	J. Biol. Chem., 193, 265(1951)	0.05%
Protein (Kjeldahl)	(Metcalf & Eddy)	0.05%
Protein (Gel Permeation Chromatography)	Analyzed on BioGel P10 Column	-----
Lactose	Biochem, J., 45, 445(1949)	0.1%
pH	Standard pH Meter	0.05 pH unit
Total Acidity	Titration, Standard Methods	0.05%
Total Solids	Gravimetric at 105°C	0.05%
Viable Bacteria	Standard Plate Count at 37°C	50 org/ml

$$Y(\%) = \frac{C_C \cdot V_C}{C_F \cdot V_F} \times 100 \qquad (2)$$

where C_C and V_C are the protein concentration in and volume of the concentrate, and C_F and V_F are the corresponding values for the feed whey.

Although yield is the important quantity in evaluating process performance, retention is a more meaningful way to examine membrane properties. For this reason, the data are presented in the form of retention. This is also a more convenient form, since it eliminates considerations pertaining to the degree of volumetric concentration of the feed material.

Protein Retention. In batch concentrations the Lowry protein retention was determined as a function of the protein content of the feed. Data are given in Figure 6 for five tests. Protein retention increased rapidly with increased feed concentration. This is due to the fact that the Lowry protein analysis included both true protein and non-protein nitrogen. As the feed became more and more concentrated the proportion of true protein, and consequently the Lowry protein retention, increased.

For all runs performed over a six-month period, protein retention remained unchanged.

The "true" protein retention of the membranes was determined by gel permeation chromatography. Feed and permeate samples were fractionated on a Biogel P-10 column (column diameter 1.5 cm, and column length, 90 cm). Ultraviolet absorbance of the column effluent was monitored as a function of effluent volume. Data are shown in Figure 7. The first peaks eluted from the column corresponded to proteins, which were excluded from the porous packing. Protein peaks were identified by calibrating the column with pure samples of soluble β-lactoglobulin and α-lactalbumin. The later peaks were other UV absorbing materials. The traces in Figure 7 are for feed whey and its corresponding permeate. It is immediately apparent that peaks corresponding to the higher molecular weight globulins, β-lactoglobulin, and α-lactalbumin, were absent in the permeate sample, except for a very small lactalbumin peak. On an area basis, about 98.5% of the protein was retained. Similar traces have

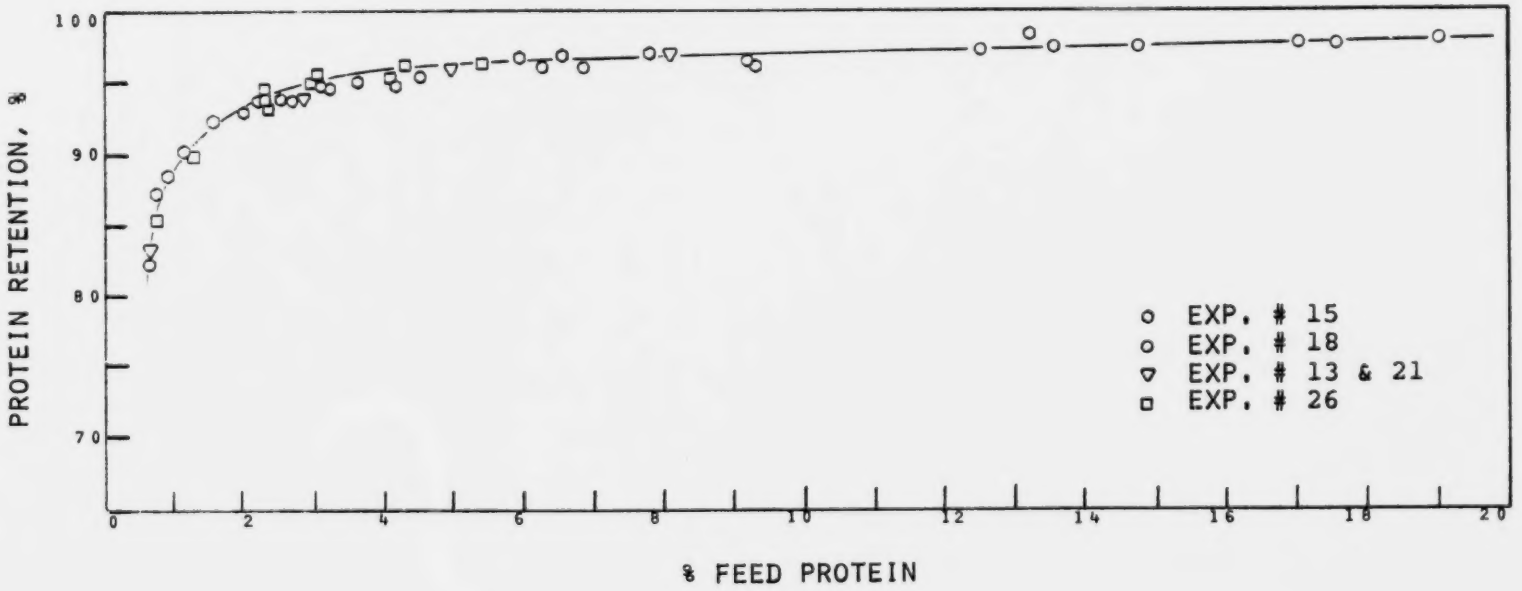


FIGURE 6

ULTRAFILTRATION DATA: PROTEIN RETENTION

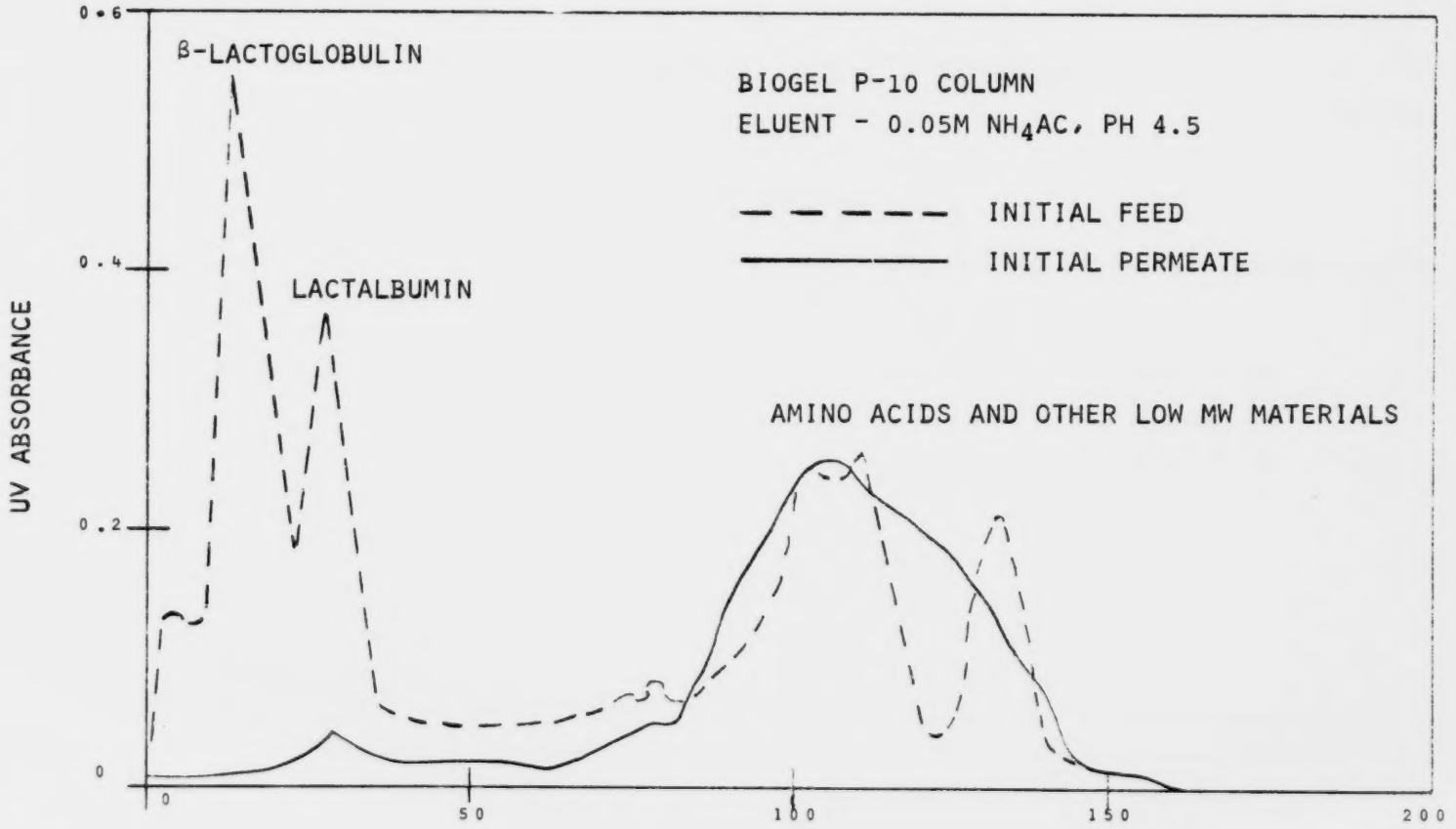


FIGURE 7

ULTRAFILTRATION DATA: GEL PERMEATION CHROMATOGRAPHY RESULTS

been generated for whey protein concentrates and corresponding permeates. These show similar behavior, with protein retentions of 98 to 99%.

Both the Lowry protein retention data and the gel permeation chromatography data indicated protein retention with HFA-180 membranes was satisfactorily high. On this basis, no further membranes were evaluated for the UF step.

Retention of Other Whey Solutes. Rejection of other solutes in the ultrafiltration experiments was very low. The data have been reduced to the form presented in Table 3.

TABLE 3

RETENTION OF LOW MOLECULAR WEIGHT SOLUTES IN ULTRAFILTRATION

Component	% Retention
non-protein nitrogen	≈0
lactose	0
salts	0
lactic acid	10-20%

Retention of non-protein nitrogen, lactose and salts was approximately 0%, and for lactic acid, very low. Thus, the membranes used for the prototype experiments very effectively retained protein while passing lower molecular weight solutes. This resulted in the production of very high protein content products through whey concentration to high volumetric reduction ratios. Data are shown in a reduced form in Figure 8. Composition of the protein concentrate on a dry solids basis is shown as a function of the degree of water removal. At water removal levels approaching 98%, protein concentrate streams can be generated with 80% protein content on a dry basis. Concentrates of this composition were generated in the course of prototype tests.

Membrane Flux. A key economic parameter in determining capital and operating costs for membrane processes is the rate of solution transport through the membrane (flux). For the ultrafiltration of proteins and other macromolecular solutions flux has been correlated with certain system parameters, expressed in Equation 3.

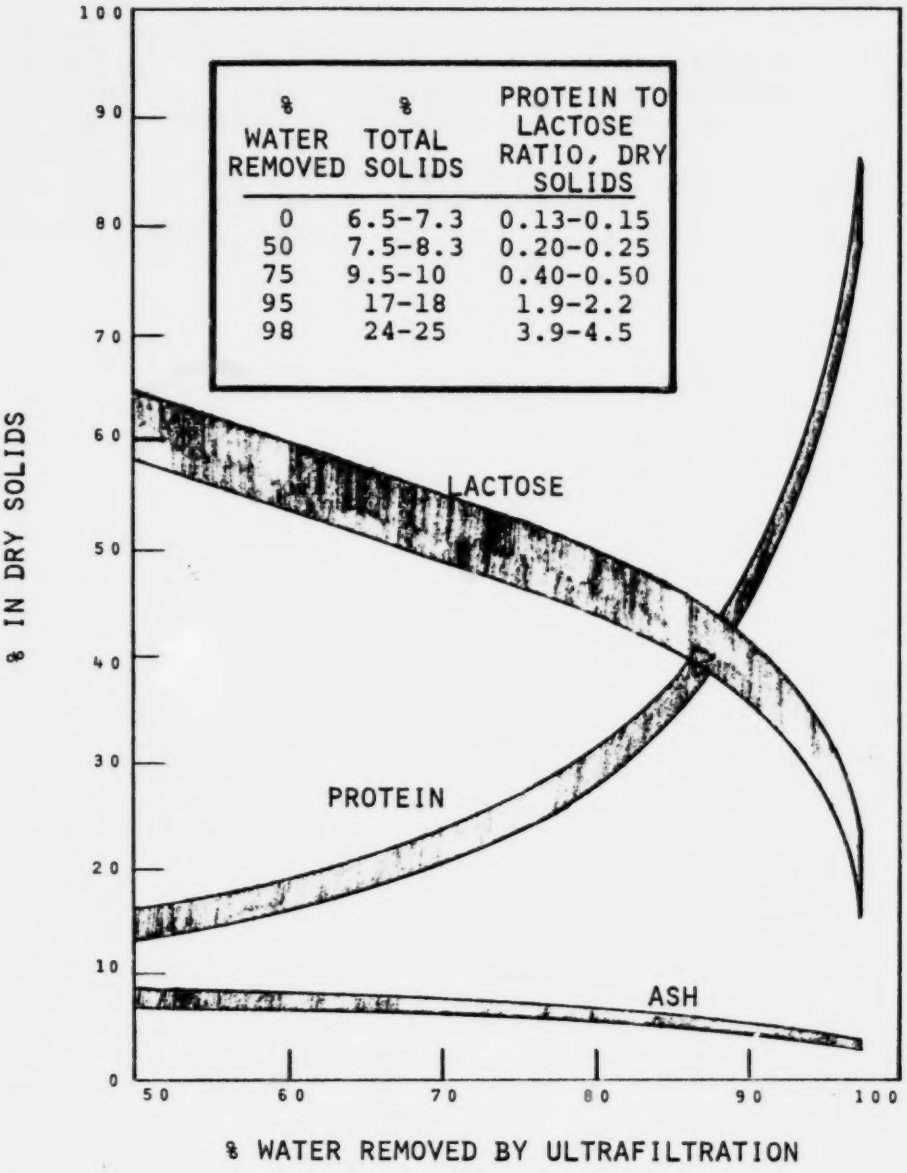


FIGURE 8

COMPOSITION OF WHEY PROTEIN CONCENTRATE

$$J = k \ln \left(\frac{C^* - C_P}{C_F - C_P} \right) \qquad (3)$$

This relationship[5] relates flux (J) to the mass transfer coefficient in the membrane-flow system (k), and certain concentration terms. C^* is a concentration determined experimentally, C_F and C_P are feed and permeate concentrations. The quantity $(C_F - C_P)$ is the "retained" protein concentration.

Flux is expected to increase in proportion to the mass transfer coefficient in the membrane system and to decrease in proportion to the log of the retained protein concentration.

In a turbulent-flow membrane system, the type of system under consideration here, the mass transfer coefficient can be correlated with additional system parameters. A generally accepted correlation is given in Equation 4.[5]

$$k = 0.0091 \left(\frac{\nu}{d} \right) Re^{0.91} Sc^{-0.65} \qquad (4)$$

The mass transfer coefficient is related to the operating Reynolds number in the system (Re), the solution Schmidt number (Sc), the solution kinematic viscosity (ν), and the tubular membrane diameter (d). The Reynolds number in turn, is determined by the circulation rate in the system (G), the tube diameter, and the solution kinematic viscosity by Equation 5.

$$Re = \frac{4G}{\pi d \nu} \qquad (5)$$

The Schmidt number is the ratio of the kinematic viscosity to the protein diffusivity, (ν/D).

Equations (3) and (4) will be used to correlate flux data.

Effect of Time on Flux (short term tests). The effect of time on membrane flux was examined in experiments lasting a few days. For well-cleaned membrane systems, a small decline in flux over the first 4 to 8 hours was generally observed. This is indicated by the data in Figure 9, in which flux in gallons/day-ft^2 (gfd) is shown as a function of operating time. Data are presented from two

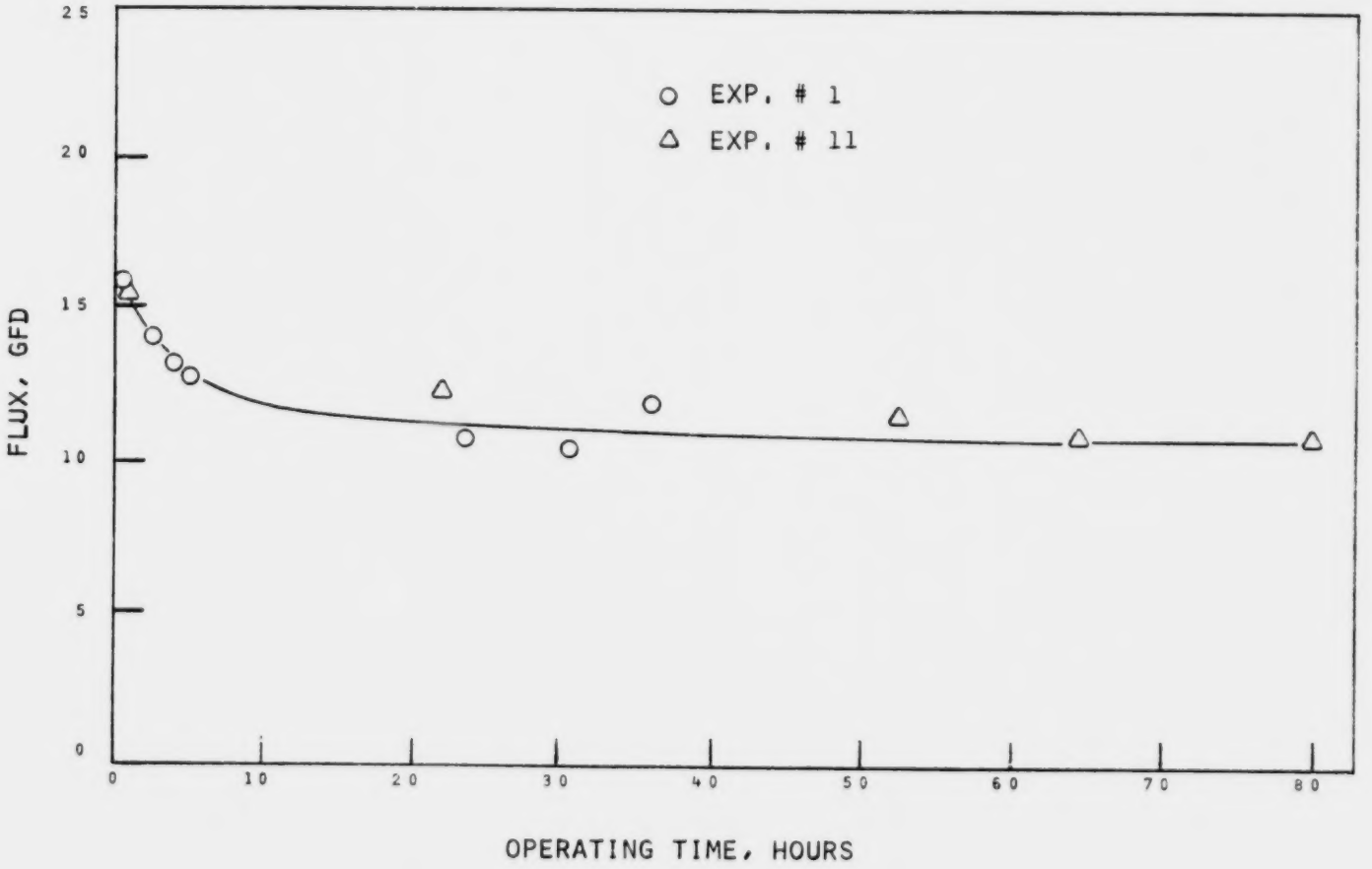


FIGURE 9

ULTRAFILTRATION DATA: DEPENDENCE OF FLUX ON TIME

(0.628 FEED PROTEIN; 60°F; 15 PSI; 18 GPM CIRCULATION RATE)

experiments. Operation was at 15 psi, 60°F, and 18 gpm circulation rate. In this differential experiment, with all permeate being returned to the feed reservoir, the Lowry protein content of the whey was maintained at a level of 0.6%.

It was not determined if the decrease in flux was due to deterioration of the whey in the membrane system, possibly related to the growth of microorganisms, or the formation of a protein precipitate on the membrane surface. However, this short-term flux decay with time was observed in both prototype and pilot-plant experiments.

Regardless of the cause of this decline the cleaning procedure developed in the course of prototype experiments was satisfactory in that initial whey fluxes could always be recovered after cleanup.

Effect of Whey Circulation Rate. According to Equations (3) and (4), UF flux is expected to increase with the 0.9 power of circulation rate. This is confirmed by the data shown in Figure 10. Flux in gfd is shown as a function of circulation rate (gpm), for both feed whey (0.6% protein) and about 6-fold concentrate (3.3% protein). The lines drawn through the data for both experiments have slopes of approximately 0.9, in excellent agreement with theory.

Effect of Operating Pressure. According to Equation (3), flux should be independent of operating pressure, since it is limited by fluid-phase mass tranfer in the membrane system and not membrane resistance. This has been confirmed experimentally and typical data are shown in Figure 11. Over the pressure range of 20 to 40 psi, flux was independent of the operating pressure.

Effect of Temperature. The major effect of temperature will be on the mass transfer coefficient k. An increase in temperature will decrease the whey viscosity and increase the protein diffusivity in solution. These two effects will lead to an increase in the mass transfer coefficient. Data illustrating this are shown in Figure 12. Flux has been plotted against the reciprocal of temperature in degrees Kelvin. The data cover the temperature range of 60 to 110°F. This Arrhenius plot is linear and lines drawn through the data correspond to activation energies of 5.1 and 5.5 kcal/mole.

For flux considerations, an optimum operating temperature

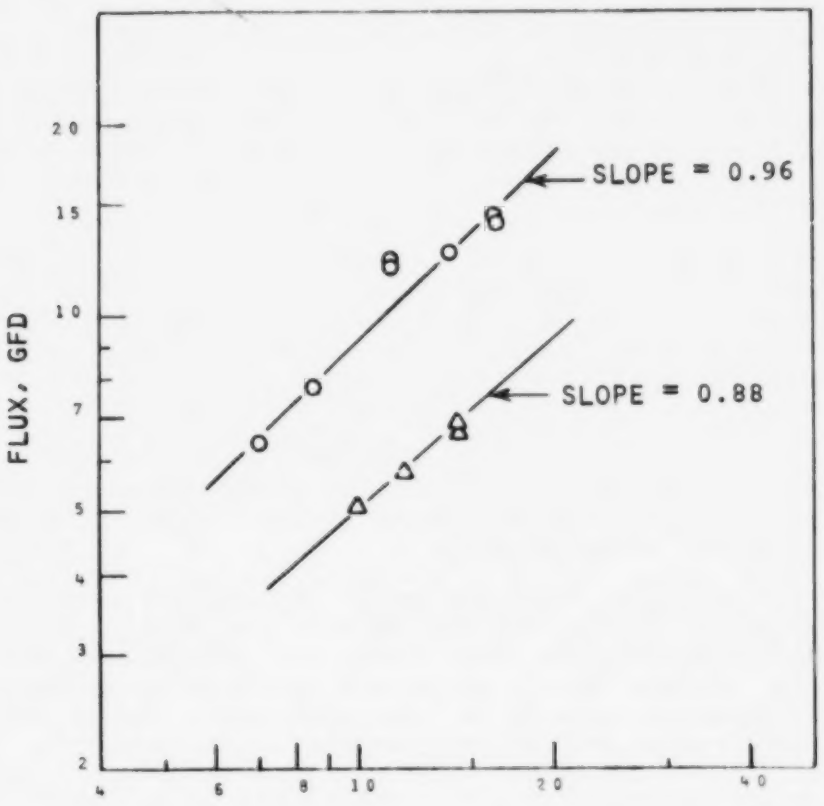


FIGURE 10

ULTRAFILTRATION DATA:
DEPENDENCE OF FLUX ON CIRCULATION RATE

○ EXP. # 7; 0.6% FEED PROTEIN;
60°F; 20 PSI

△ EXP. # 11; 3.3% FEED PROTEIN;
79°F; 12 PSI

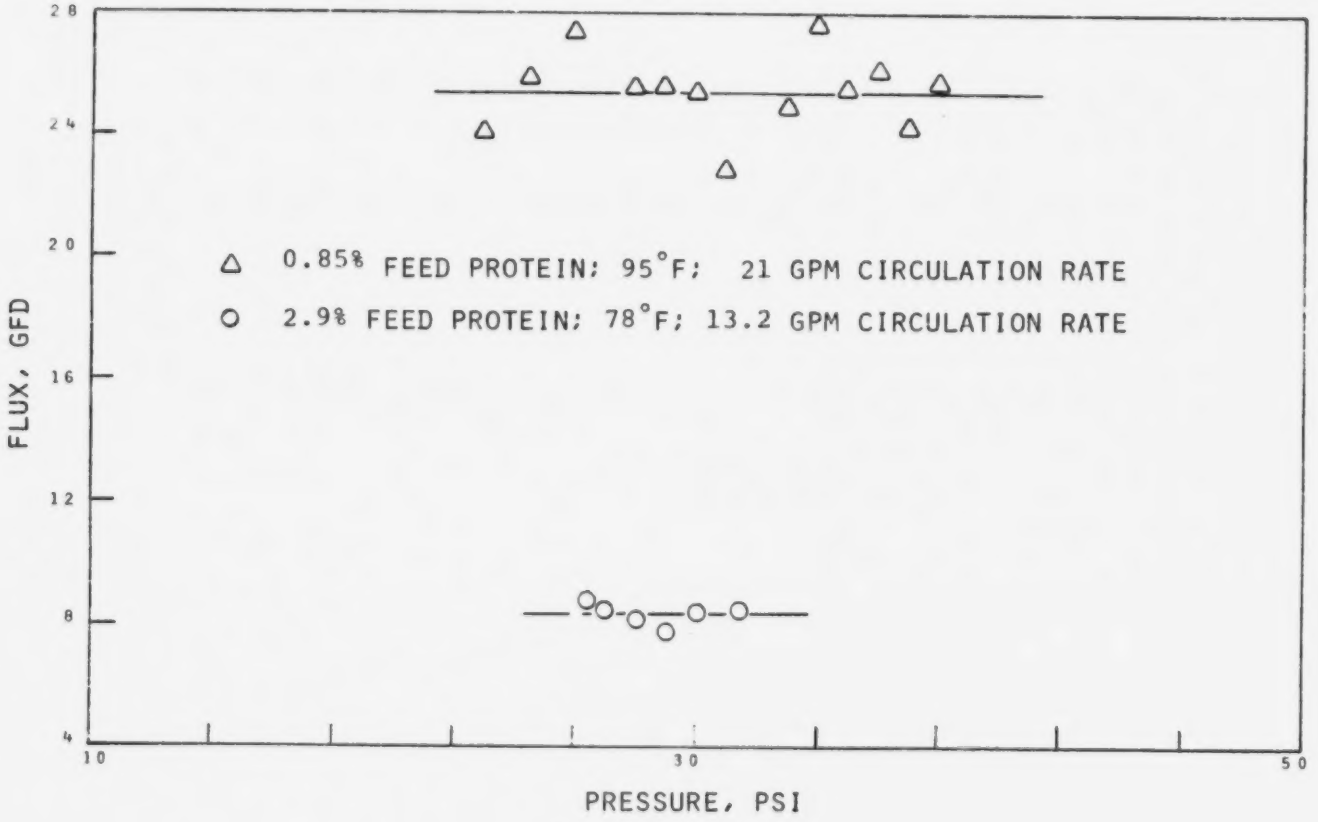


FIGURE 11

ULTRAFILTRATION DATA: DEPENDENCE OF FLUX ON PRESSURE

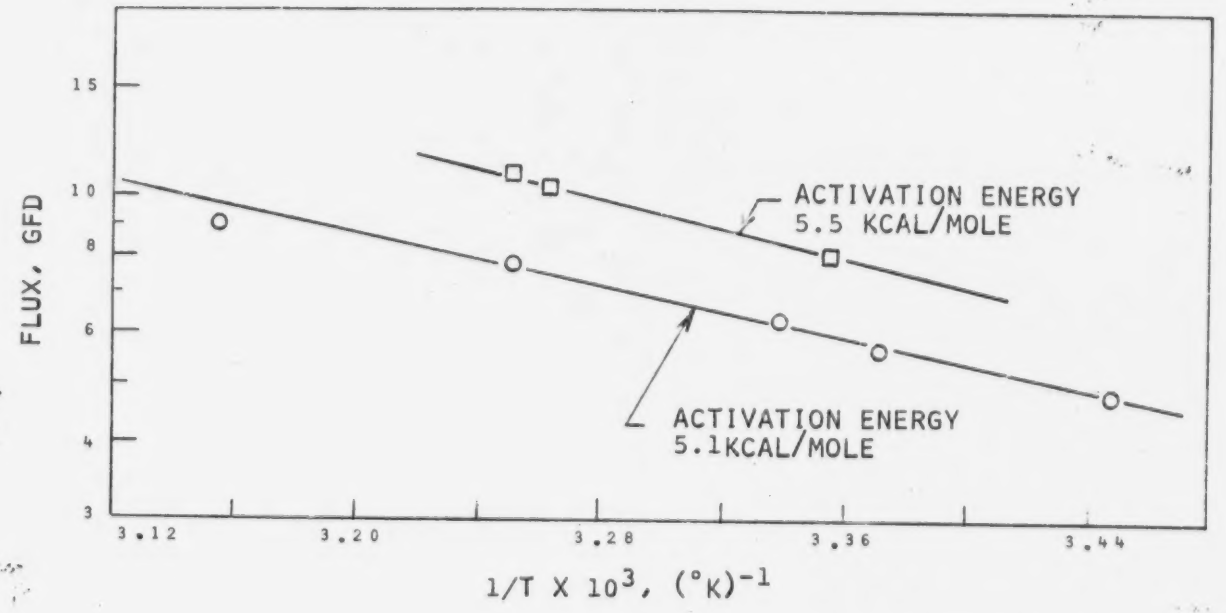


FIGURE 12

ULTRAFILTRATION DATA: DEPENDENCE OF FLUX ON TEMPERATURE

❑ EXP. # 15; 3.2% FEED PROTEIN; 38 PSI; 13.2 GPM CIRCULATION RATE

○ EXP. # 11; 4% FEED PROTEIN; 12 PSI; 14.3 GPM CIRCULATION RATE

appears to be about 125°F. For cottage cheese whey this minimizes plant heat exchange requirements since whey is received hot from the cheese vats. Preliminary results, not described in this report, show little if any heat denaturation of the whey proteins at this temperature. Furthermore, as will be discussed, the rate of bacterial growth at 125°F appears to be low.

Effect of Concentration. According to Equation (3), flux is expected to decrease logarithmically with increasing feed concentration.

Data shown in Figure 13 support this hypothesis. Flux is plotted versus log of the retained Lowry protein concentration for operation between 61°F and 120°F. Although there is some scatter, the data can be fit by straight lines on this semi-log plot. As is expected, increased temperature resulted in increased flux levels. From the intercept of the lines, the quantity (C^*-C_p) in Equation (3) is estimated to be 30%.

Life Tests. Life tests have been in progress with 14 tubular membranes since September, 1969. Data for flux and protein retention over the ensuing 14 months are shown in Figure 14.

Standard procedure has been to operate with a single batch of whey for one week, followed by clean-up (see page for procedure) and operation with a new batch. Fluxes recorded were measured within 10 to 24 hours after introduction of fresh whey. In the course of operation, over a 5 to 7 day period, substantial microbiological growth was observed in the membrane system. This was manifested in very high counts of yeasts, molds, and thermophiles. In spite of this very extensive and regular microbiological contamination, no detectable deterioration of the membranes occurred.

Flux for raw whey, with or without filtration, averaged about 20.5 gfd, while membrane protein retention varied between 80 and 90%. Scatter in protein retention is probably due to variation in the cheese whey. It is apparent that no detectable deterioration occurred in either membrane flux or protein retention, demonstrating membrane life in excess of one year. These results illustrate the dependability of the low-pressure ultrafiltration system, and have been confirmed in the pilot plant operation.

During the life tests, the membranes were exposed to whey

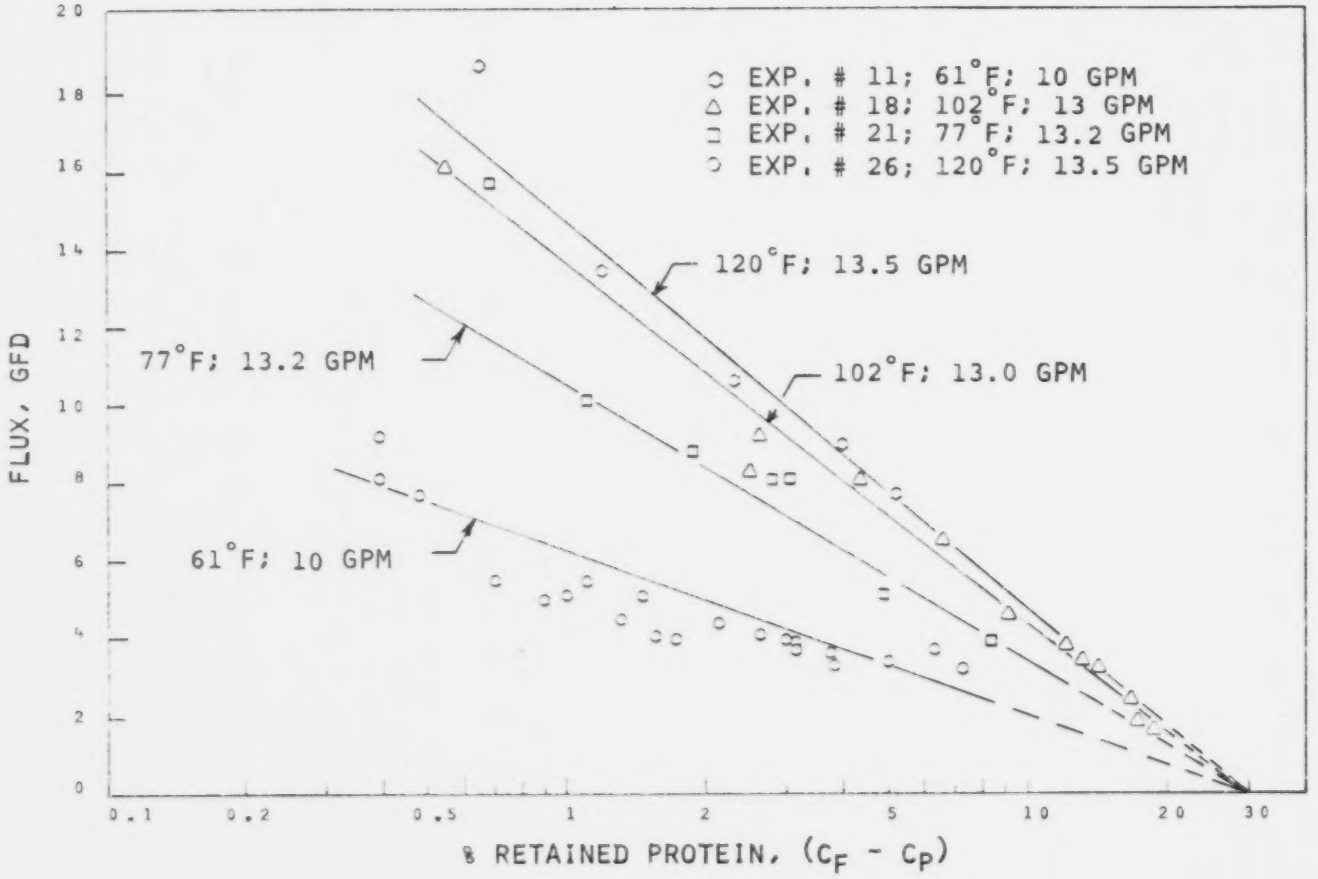


FIGURE 13

ULTRAFILTRATION DATA: DEPENDENCE OF FLUX ON PROTEIN CONCENTRATION

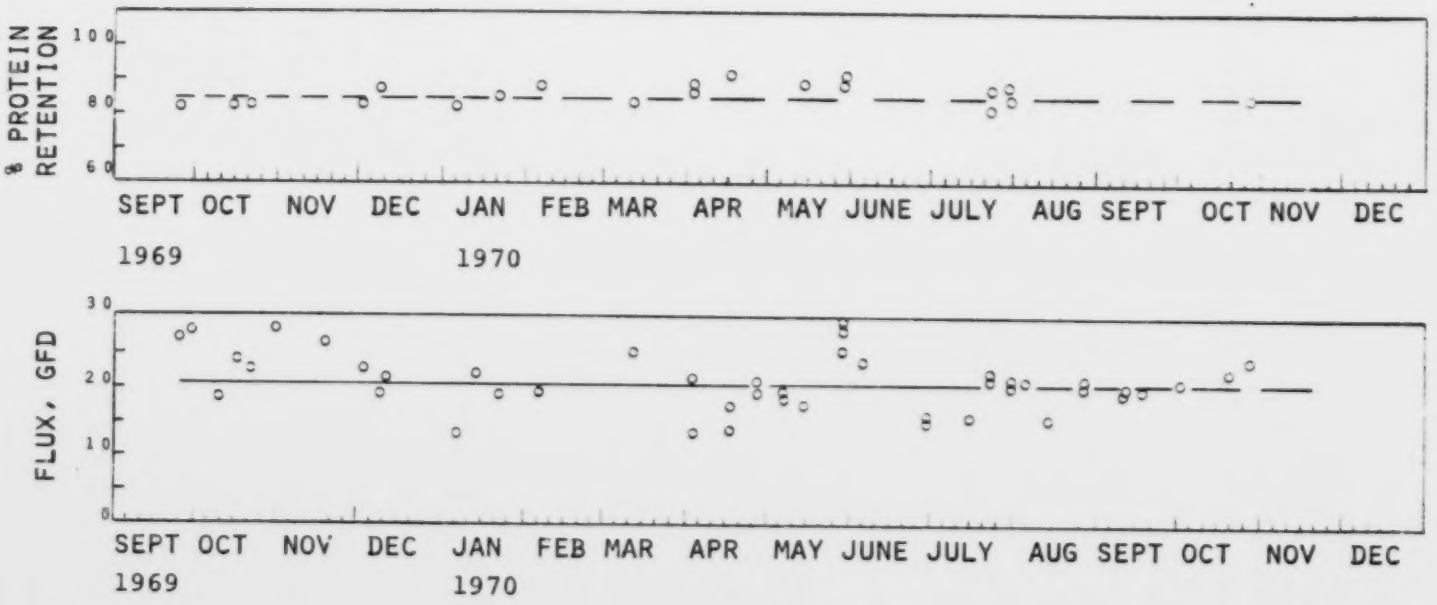


FIGURE 14

ULTRAFILTRATION LIFE DATA: FLUX AND PROTEIN RETENTION

(COTTAGE CHEESE WHEY WITH AND WITHOUT FILTRATION AS FEED; 0.8% PROTEIN; 15 GPM CIRCULATION RATE; 113°F; 25 PSI)

under operating conditions slightly more than 50% of the time. The remaining time was taken up by water tests, system cleaning and sanitizing, shutdown due to non-availability of whey, or for vacations and holidays. During the life tests, there was no case of membrane failure.

Cleaning and Sanitizing.

Procedures. In the course of the long-term experiments, it became apparent that membrane fouling occurred. This was evidenced by formation of "scale" on the membrane surfaces, resulting in reduced water and whey fluxes. In addition, microbiological contamination of the system became severe in the absence of cleaning and sanitizing. For these reasons, a variety of dairy cleaners and sanitizers were examined. Important considerations were the following:

- -ability of cleaners to remove scale from the membranes, resulting in recovered water flux;
- -absence of chemical attack of the membranes by cleaning solutions;
- -effective sanitization of the membranes by sanitizers;
- -absence of chemical attack of the membranes by sanitizers.

A variety of acid, neutral, and alkaline detergents, both ionic and non-ionic, were examined. In addition, several sanitizing agents, including formaldehyde, iodofores, hydrogen peroxide, and chlorine, were tested. Although many of the cleaners and sanitizers were effective, the following cleaning and sanitizing sequence was adopted early in the life tests program because of its simplicity, effectiveness, and acceptability to the dairy industry.

Cleaning. Following operation with whey, the membrane system was flushed with tap water. Next, a cleaning solution consisting of 1 oz Alcozyme per 2 gals of water was circulated through the membrane system at 40-50^{o}C. Alcozyme is an alkaline enzyme detergent manufactured by the Alconox Co., N.Y. The pH of the cleaning solution was adjusted to 9.1-9.3, if originally above this level. Adjustment was made with HCl. The cleaning solution was circulated through the membrane system at 15-25 gpm, for 30 min to 1 hr. During this period, permeate was returned

to the cleaning solution tank. Following cleaning, the membrane system was again flushed with tap water, and stored filled with water until the next experiment. If storage was to be longer than one week, the system was sanitized prior to filling with water.

Sanitizing. Prior to operation with whey the membrane system was sanitized. A solution of Antibac B (Wyandotte Chemical Co.) at a concentration of 1 oz/12 gal water was circulated through the membranes. This sanitizing solution provides 100 ppm free chlorine at a pH of about 5. The sanitizing solution was circulated for 15-20 minutes at 80-100°F and 10-20 gpm. As with the cleaning solution, the permeate was returned to the santizing solution tank. Following this operation, the sanitizing solution was displaced from the membrane system with whey. Ultrafiltration experiments commenced as soon as displacement was complete.

Microbiological Data. The effectiveness of the above cleaning and sanitizing procedure is indicated by the following data.

In Figure 15 total plate counts of one batch of whey are presented as a function of holding time in the membrane system. The actual counts correspond to counts in samples taken as concentration proceeded during a batch experiment. Starting at a level of slightly more than 1,000 org/ml, the total plate count increased to about 80,000 org/ml. Ultrafiltration membranes selectively retain microorganisms (microorganisms have orders-of-magnitude higher molecular weights than whey proteins), and these counts should be corrected for the volumetric concentration ratio to have a measure of actual growth rates. This has been done as indicated. The actual growth rate was indeed low.

In Table 4 are shown microbiological data for cottage cheese whey protein produced in the prototype batch concentration system. A fluid protein concentrate was produced containing about 15% protein (70% protein on dry solids basis). Total plate count of the concentrate was 74,000 org/ml. In freeze-drying the protein, this dropped to 37,000 org/gm; and in spray drying the protein, this dropped to 600 org/gm. Apparently thermal conditions in the spray drier were effective for killing bacteria. The complete absence of yeasts, molds, and coliforms is to be noted.

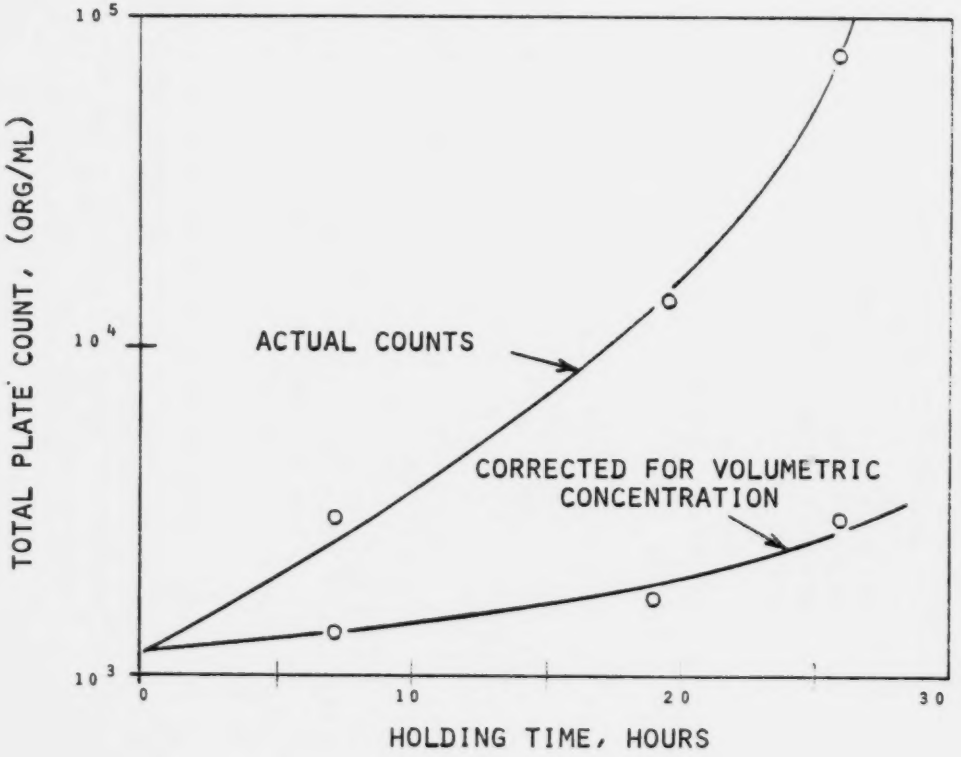


FIGURE 15

ULTRAFILTRATION DATA: BACTERIAL GROWTH DURING WHEY CONCENTRATION

Although the prototype system was of a non-sanitary design, products were generated with microbiological counts acceptable for food-grade products. This result has subsequently been reproduced in the pilot plant operation.

TABLE 4

MICROORGANISMS IN COTTAGE CHEESE WHEY PROTEIN
(77% Protein in Dry Solids)

	Fluid Concentrate	Freeze Dried Protein	Spray Dried Protein
Total Plate Count (TSY Agar)	74,000 org/ml	37,000 org/ml	600 org/ml
Yeasts and Molds (Sabourauds Dextrose Agar)	---	0	0
Coliforms (McConkey's Agar)	---	0	0

REVERSE OSMOSIS

Experimental Procedure and Results

Apparatus and Procedure. Two prototype systems were used for RO experiments, similar in layout to that of the UF systems (See Figure 5). Major differences include the use of high-pressure pumps, valves and piping since operation was at substantially higher pressures. Positive-displacement piston pumps were used, with stainless steel accumulators added to damp out flow pulsations. Although major pulsations were not present "micropulsations" were, are could have contributed in some degree to module failures discussed below.

Experiments were of two types, differential and batch concentration. In differential tests the effects of pressure and recirculation rate on flux and membrane rejection were examined. In batch concentration experiments the effect of feed concentration on membrane flux and rejection was determined. A variety of membrane modules were examined, including systems manufactured by Havens International, American Standard, Universal Water Corporation, and Aerojet General. The number and types of modules are listed in Table 5 (page 53).

Analytical Procedures. The analytical procedures which were used have been discussed in the section entitled "Prototype Experiments: Ultrafiltration." These included analyses of feed and permeate samples for COD, total solids, lactic acid and lactose. All of the experiments, unless otherwise noted, were performed with UF permeate as feed.

Membrane Rejection Properties. Rejection characteristics have been determined for the modules listed in Table 5. These data are presented in this section.

Solids rejection and solids level in the permeate are shown as functions of feed concentration in Figure 16.

Data are given for several types of modules including Havens Types 300 and 310, American Standard TM2-4 with AS-189 membranes, American Standard TM5-14 modules with AS-194 and AS-197 membranes, Universal Water Corp. modules, and eight Aerojet tubular membranes. Total solids rejection was observed to be best for the Aerojet tubes and the AS-194 membranes, followed by the Universal Water Corp. modules. The AS-197 membranes did not exhibit as high a rejection as would be expected since these membranes should have higher retention properties than the AS-194 membranes. Havens Type 300 and 310 modules showed the lowest rejection.

Similar data for COD rejection are given in Figure 17. COD rejection paralleled that for total solids. The most retentive membranes, the American Standard AS-194 membranes and Aerojet tubes, had COD rejections exceeding 99%, and this is an obtainable level for plant operation.

Data for lactose rejection are given in Figure 18. Although there was more scatter for these data, rejection of lactose by the membrane modules was of the same order as total solids and COD rejections.

The rejection of lactic acid by RO membranes was only moderate. This is demonstrated by the data of Figure 19. Rejection is shown as a function of lactic acid concentration in the feed. Typical rejection levels ranged between 80 and 90% with UF permeate as feed. When raw cottage cheese whey was used as feed, rejection levels were somewhat higher. This cannot be easily explained, but may be due to the formation of a high-rejection dynamic membrane by whey proteins.

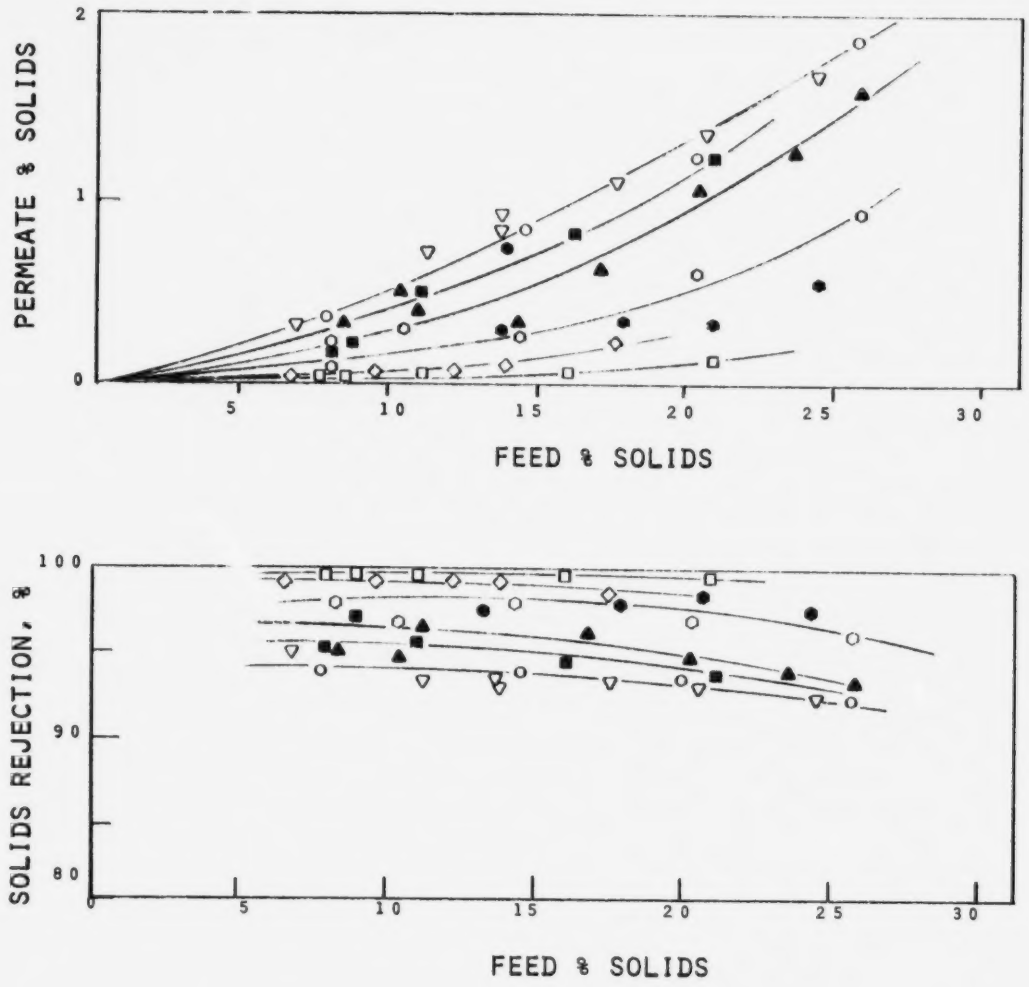


FIGURE 16

REVERSE OSMOSIS DATA: PERFORMANCE OF DIFFERENT MODULES

(COTTAGE CHEESE WHEY - UF PERMEATE AS FEED)

- ▽ HAVENS 310 MODULE, 575 PSIG
- ● UNIVERSAL WATER CORP. MODULE #349, 600 PSIG
- ○ AMERICAN STANDARD AS-189, 710PSIG
- ■ AMERICAN STANDARD AS-197, 570 PSIG
- ▲ HAVENS 300 MODULE, 740 PSIG
- ○ UNIVERSAL WATER CORP. MODULE #395, 780 PSIG
- ◇ AEROJET TUBES, 590 PSIG
- □ AMERICAN STANDARD AS-194, 580 PSIG

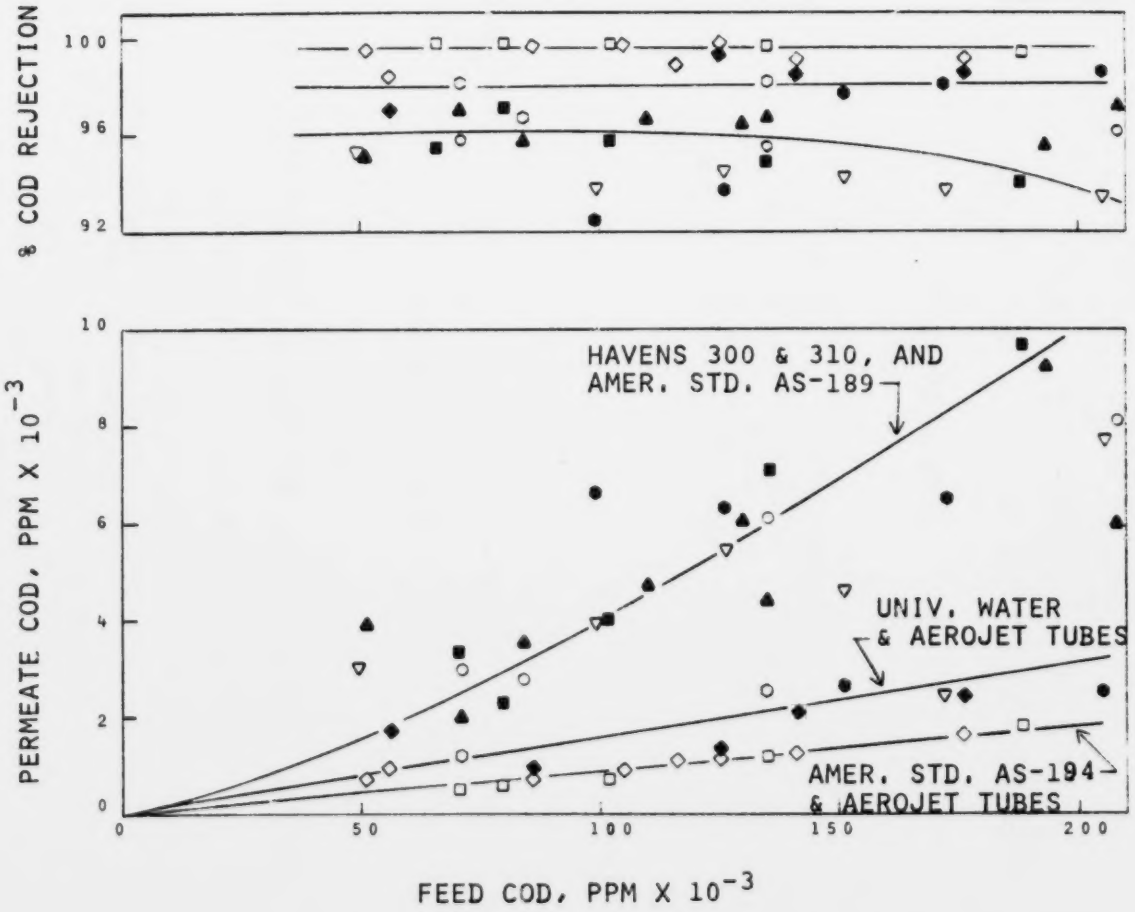


FIGURE 17

REVERSE OSMOSIS DATA: COD REJECTION

(SEE FIGURE 16 FOR LEGEND; EXCEPT ◆ 590 PSI, AEROJET TUBES)

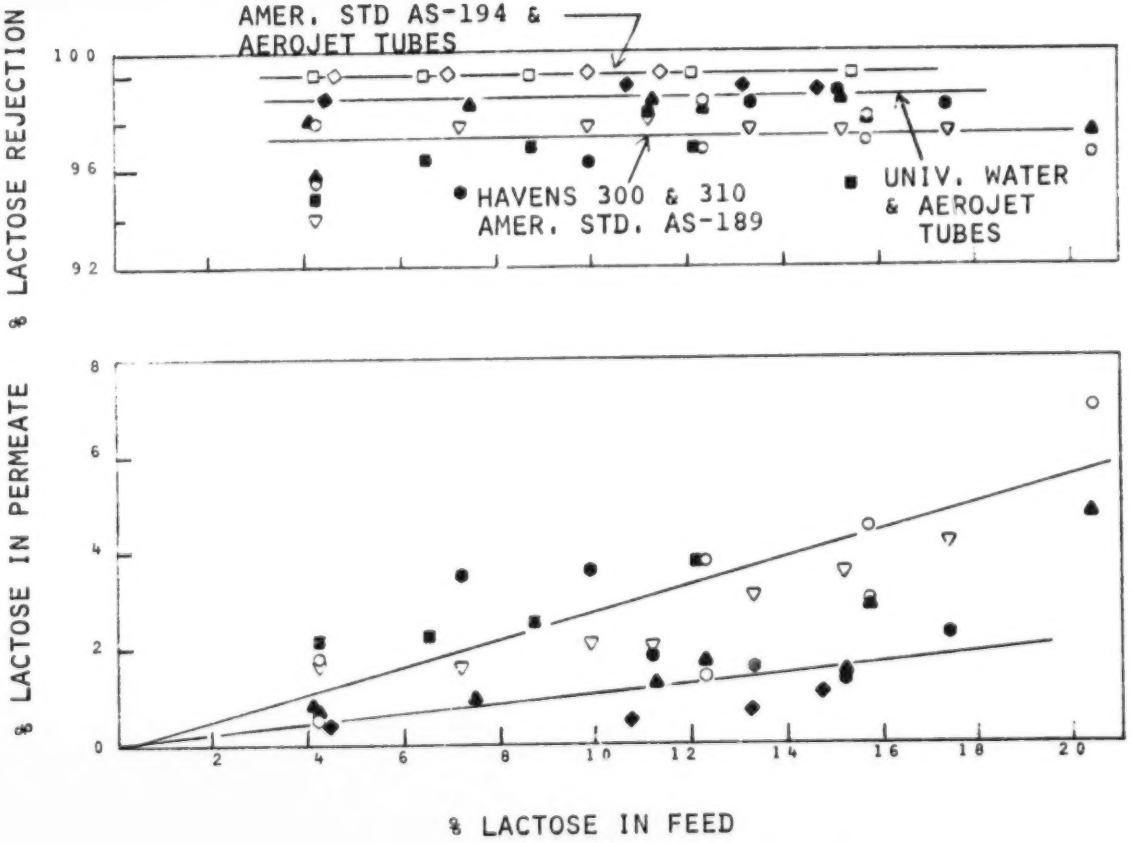
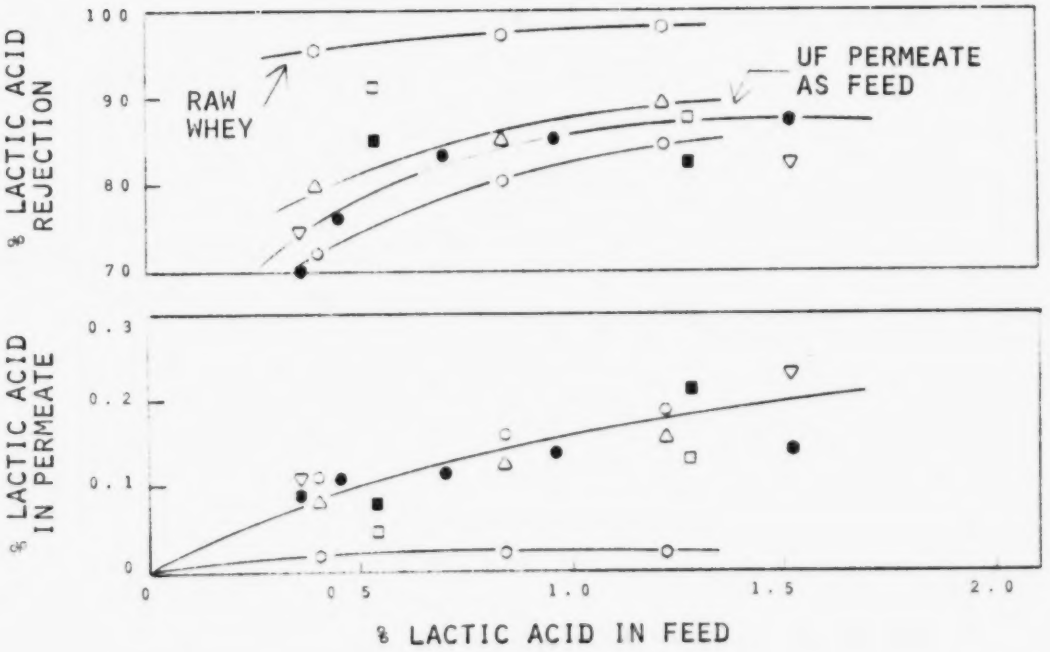


FIGURE 18

REVERSE OSMOSIS DATA: LACTOSE REJECTION

(SEE FIGURE 16 FOR LEGEND; EXCEPT ◆ 590 PSI, AEROJET TUBES)



UNIVERSAL WATER CORP. MODULES

○ RAW WHEY AS FEED, 680 PSI, 26°C, 2 GPM
● UF PERMEATE AS FEED, 590 PSI, 25°C, 1.8 GPM

AMERICAN STANDARD MODULES

◑ RAW WHEY AS FEED, 610 PSI, 26°C, 2 GPM, AS-189 MEMBRANES
◡ UF PERMEATE AS FEED, 600 PSI, 25°C, 2 GPM, AS-189 MEMBRANES
– UF PERMEATE AS FEED, 570 PSI, 25°C, 1.8 GPM, AS-194 MEMBRANES
■ UF PERMEATE AS FEED, 570 PSI, 25°C, 1.8 GPM, AS-197 MEMBRANES

HAVENS MODULES

◟ UF PERMEATE AS FEED, 570 PSI, 25°C, 1.8 GPM, 310 TYPE MEMBRANES
◞ RAW WHEY AS FEED, 650 PSI, 26°C, 2 GPM, 300 TYPE MEMBRANES

FIGURE 19: REVERSE OSMOSIS DATA: LACTIC ACID REJECTION

By comparison of Figures 17 and 18 it is seen that COD rejections are lower than lactose rejections. This is attributed to the partial passage of lactic acid into the permeate. For this reason treatment of high-acid wheys will result in a higher effluent BOD. It is desirable, therefore, to treat cottage cheese whey as soon as possible after it is removed from the cheese vats.

Effect of Pressure on Membrane Rejection. According to RO theory, an increase in operating pressure will increase membrane rejection efficiency. This is demonstrated by the data in Figures 20, 21, and 22. Rejection data are shown for the American Standard TM2-4 module with AS-189 membranes. Rejections for total solids, COD and lactose are observed to increase substantially with increases in operating pressure. These data indicate that operation of the RO section should be at as high a pressure as possible in order to minimize the final effluent BOD. The major limitation will be mechanical, with the maximum operating pressure determined by two factors: membrane compaction and mechanical strength of the membrane supports.

Membrane Flux: General. Reverse osmosis systems are generally designed so that concentration polarization is not an important consideration. This is achieved by determining the circulation rate above which flux and rejection are independent of circulation rate, and operating above this level. Under this condition, membrane flux is determined by two parameters: the inherent membrane resistance to water transport and the osmotic pressure of the feed. Solution flux is given by

$$J = A(\Delta P - \Delta \pi) \tag{6}$$

where J is the flux; A is the membrane constant; and ΔP and $\Delta \pi$ are the differences in applied pressure and osmotic pressure across the membrane. The flux of dissolved materials (solutes) is described by the equation

$$J_{sol} = B\Delta C \tag{7}$$

where J_{sol} is the flux of a dissolved component, B is the permeation coefficient through the membrane, and ΔC is the concentration gradient across the membranes. These two equations predict increased rejection with increased operating pressure. This fact is a consequence of the nature of the driving forces in RO. Thus, the experimental results presented above, showing rejection increasing with pressure, are in accord with theory.

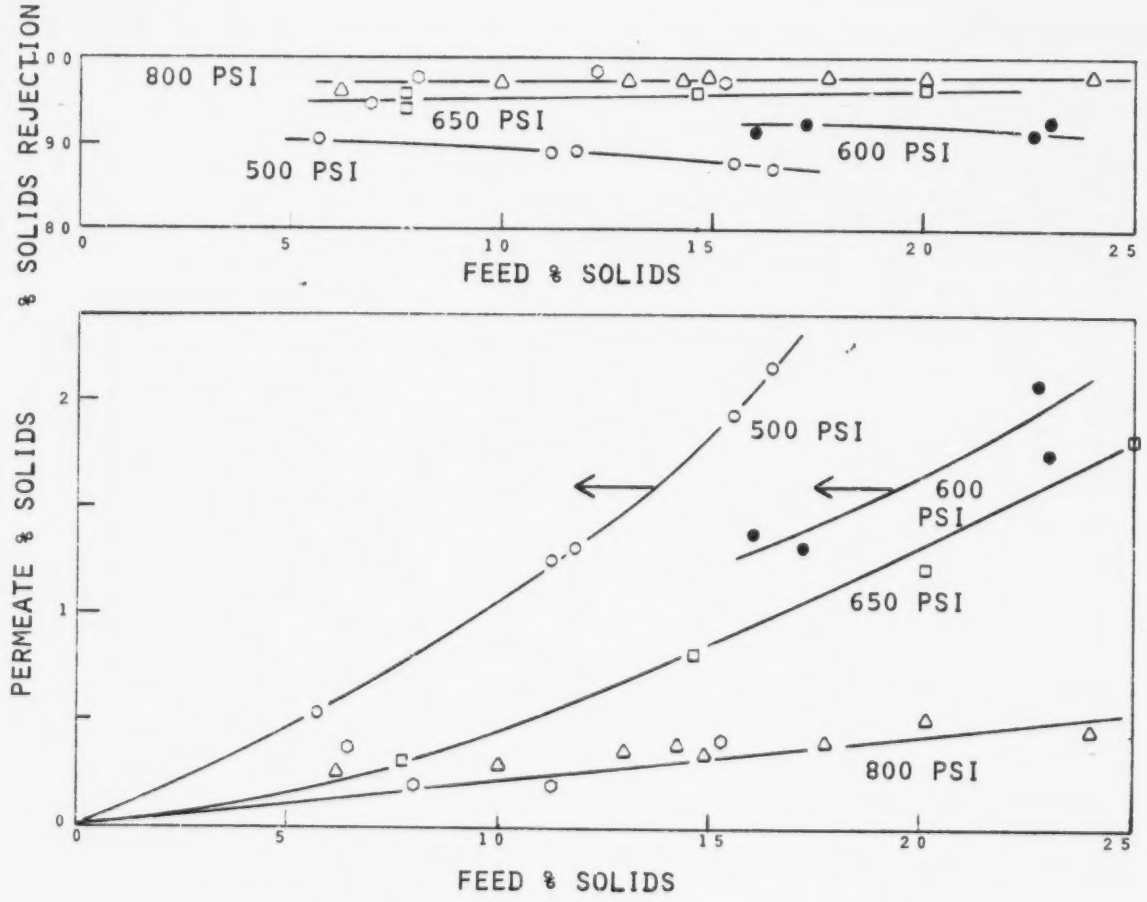


FIGURE 20

REVERSE OSMOSIS DATA: DEPENDENCE OF SOLIDS REJECTION ON PRESSURE AND CONCENTRATION

(AMERICAN STANDARD AS-189 MEMBRANES; CHEESE WHEY - UF PERMEATE AS FEED)

(○- 500 PSI; ● - 600 PSI; □- 650 PSI; △&○ - TWO TESTS AT 800 PSI)

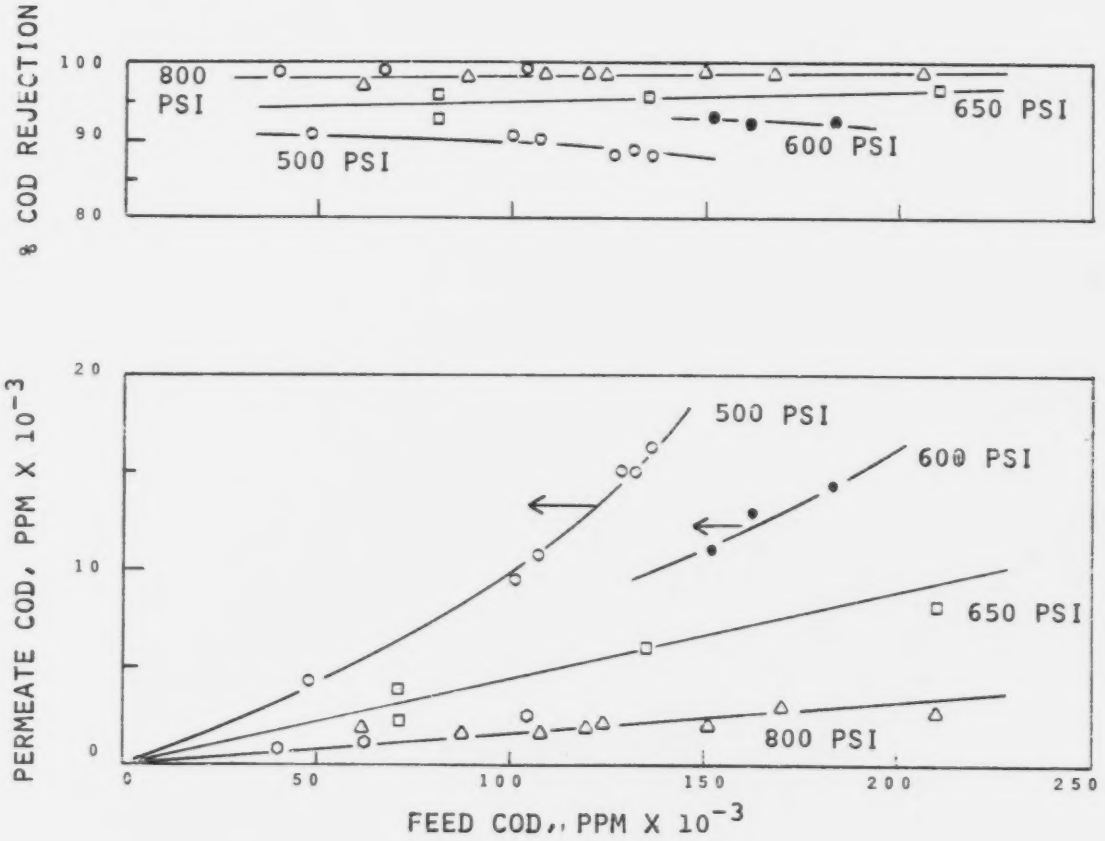


FIGURE 21

REVERSE OSMOSIS DATA: DEPENDENCE OF COD REJECTION ON PRESSURE AND CONCENTRATION

(AMERICAN STANDARD AS-189 MEMBRANES; CHEESE WHEY - UF PERMEATE AS FEED)

(○- 500 PSI; ●- 600 PSI; □- 650 PSI; △& ○ - TWO TESTS AT 800 PSI)

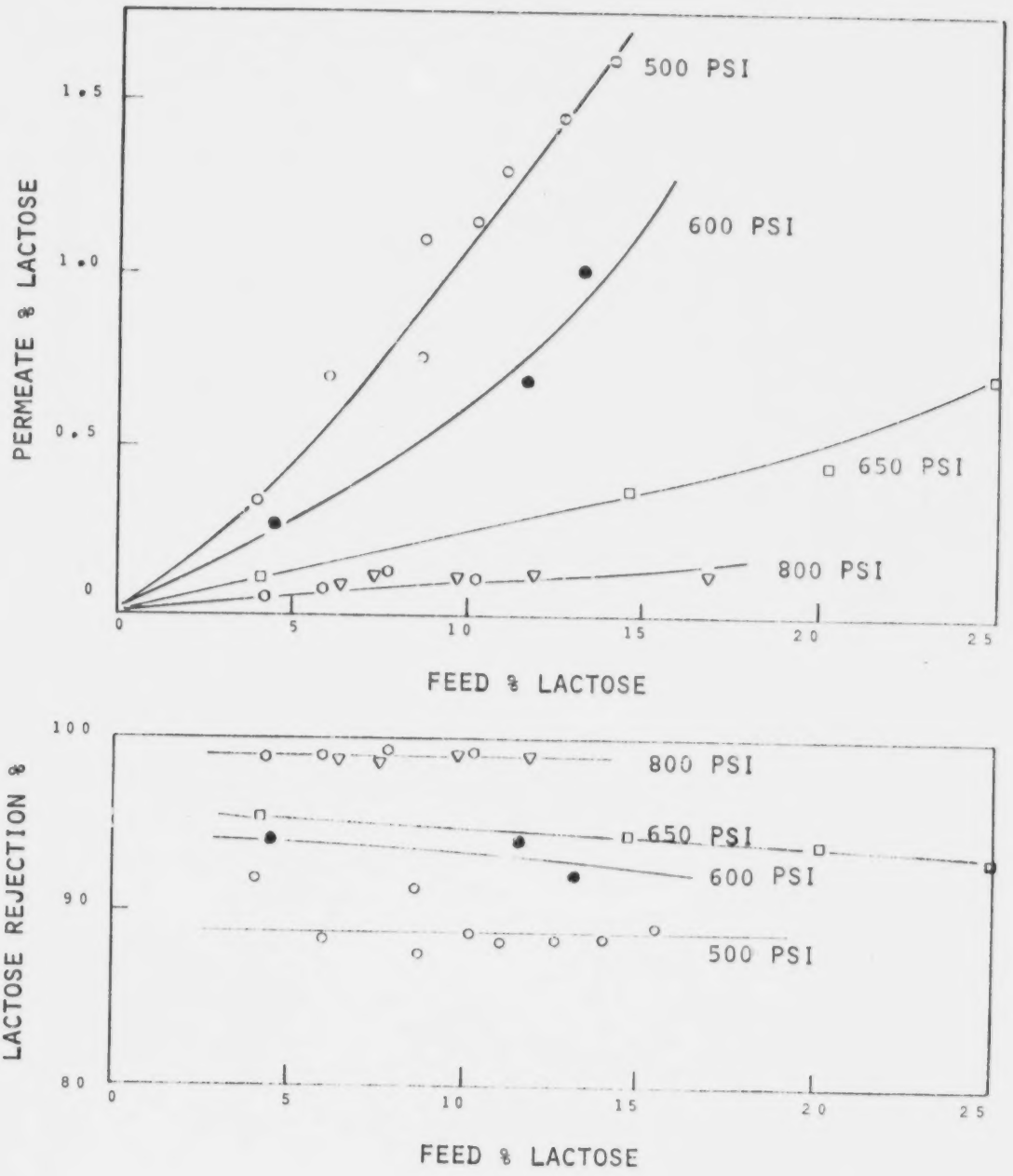


FIGURE 22

REVERSE OSMOSIS DATA: DEPENDENCE OF LACTOSE REJECTION ON PRESSURE AND CONCENTRATION

(AMERICAN STANDARD AS-189 MEMBRANES; CHEESE WHEY - UF PERMEATE AS FEED)
(○ - 500PSI; ● - 600PSI; □ - 650PSI; △ & ▽ TWO TESTS AT 800 PSI)

Dependence of Flux on Feed Concentration. Flux as a function of concentration for UF permeate is given in Figures 23, 24 and 25, for a variety of different membrane modules. Data are shown for Universal Water Corp. modules, Aerojet tubular membranes, Havens 300 and 310-Type modules and American Standard modules with AS-189, AS-194 and AS-197 membranes.

For all the modules tested, flux decreased with increasing feed concentration as predicted by Equation (6). The osmotic pressure for a 6% solids solution is approximately 100 psi. On this basis, a 24% solids solution would have an osmotic pressure of about 400 psi. Between the range of 6 and 24% solids, osmotic pressure would increase approximately linearly with concentration. The increase in osmotic pressure of the feed, therefore, accounts for the marked reduction in flux observed for all modules. Although some differences in the flux levels between modules can be explained on the basis of difference in operating pressure, it appears that the highest flux module was the Universal Water Corp. module. The other membrane systems tested had more or less the same flux, with the exception of the AS-189 module (when new). However, comparison of the flux data for the AS-189 membrane (when new) and flux data at a later date shows that membrane flux decline occurred.

Based on these data, it appears technically feasible to produce concentrates with solids in the range of 25% from UF permeate. Higher solids content concentrates can probably be produced at operating pressures exceeding the level of 600-700 psi used in these experiments.

Effect of Pressure on Flux. According to Equation (6), flux should increase with increasing operating pressure. This is confirmed by the data shown in Figure 26, for AS-189 membranes in the American Standard TM2-4 module. Flux, shown as a function of feed concentration, is observed to increase with operating pressure.

Effect of Circulation Rate on Membrane Performance. It is desirable to operate at a circulation rate (through the membrane modules) above which flux and membrane rejection are independent of circulation rate. An experiment was performed with a Universal Water Corp. module, American Standard module, and Havens Type 300 module to determine this minimum rate. All three tubular membrane systems were used without turbulence promoters or volume displacers, presently incorporated in American Standard and

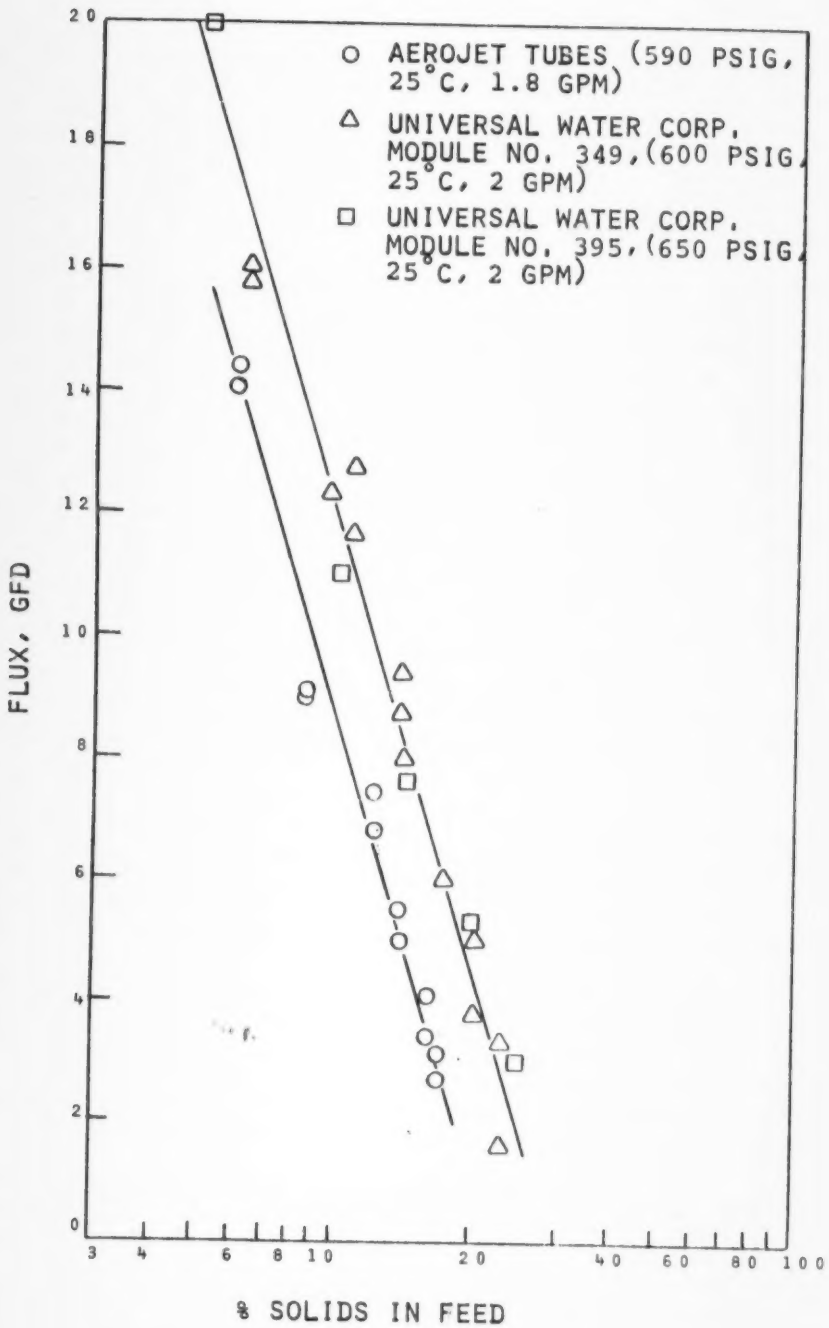


FIGURE 23

REVERSE OSMOSIS DATA: DEPENDENCE OF FLUX ON FEED SOLIDS CONCENTRATION

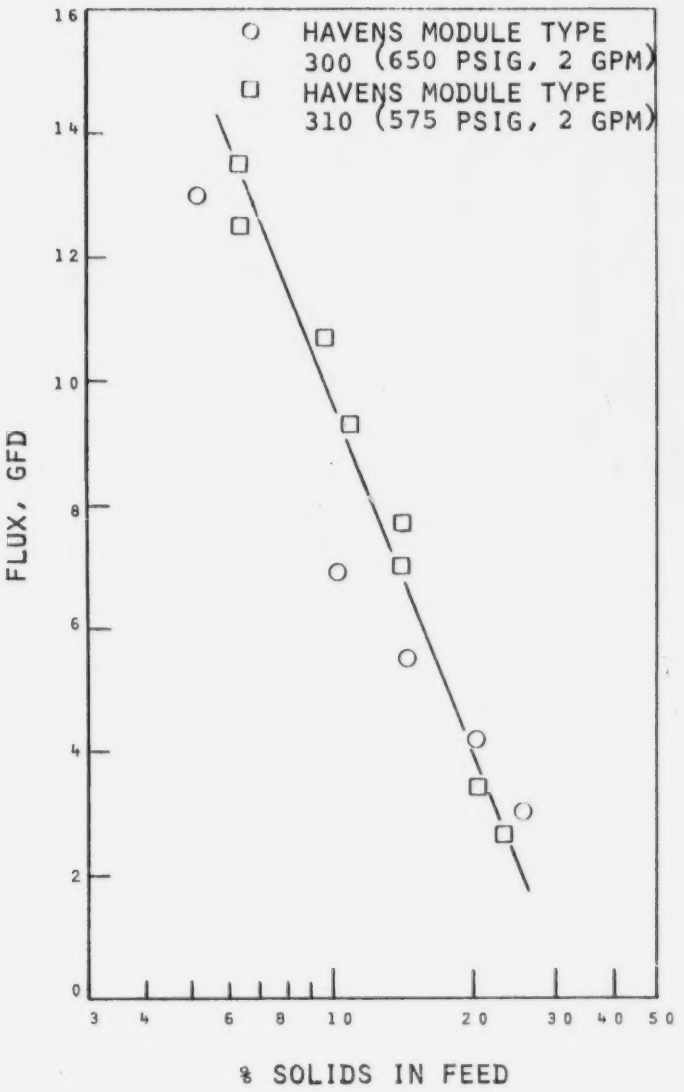


FIGURE 24

REVERSE OSMOSIS DATA: DEPENDENCE OF FLUX ON FEED SOLIDS CONCENTRATION

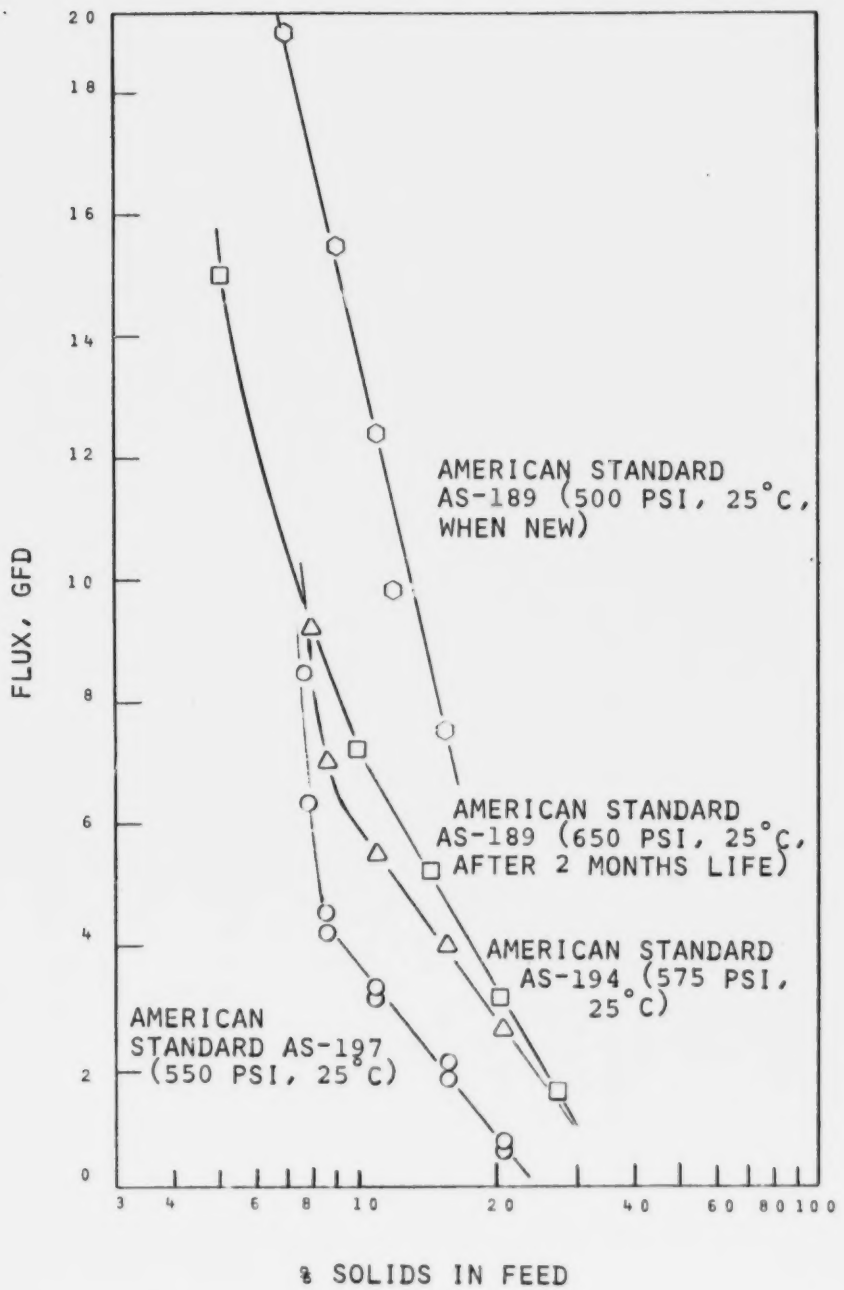


FIGURE 25

REVERSE OSMOSIS DATA: DEPENDENCE OF FLUX ON FEED SOLIDS CONCENTRATION

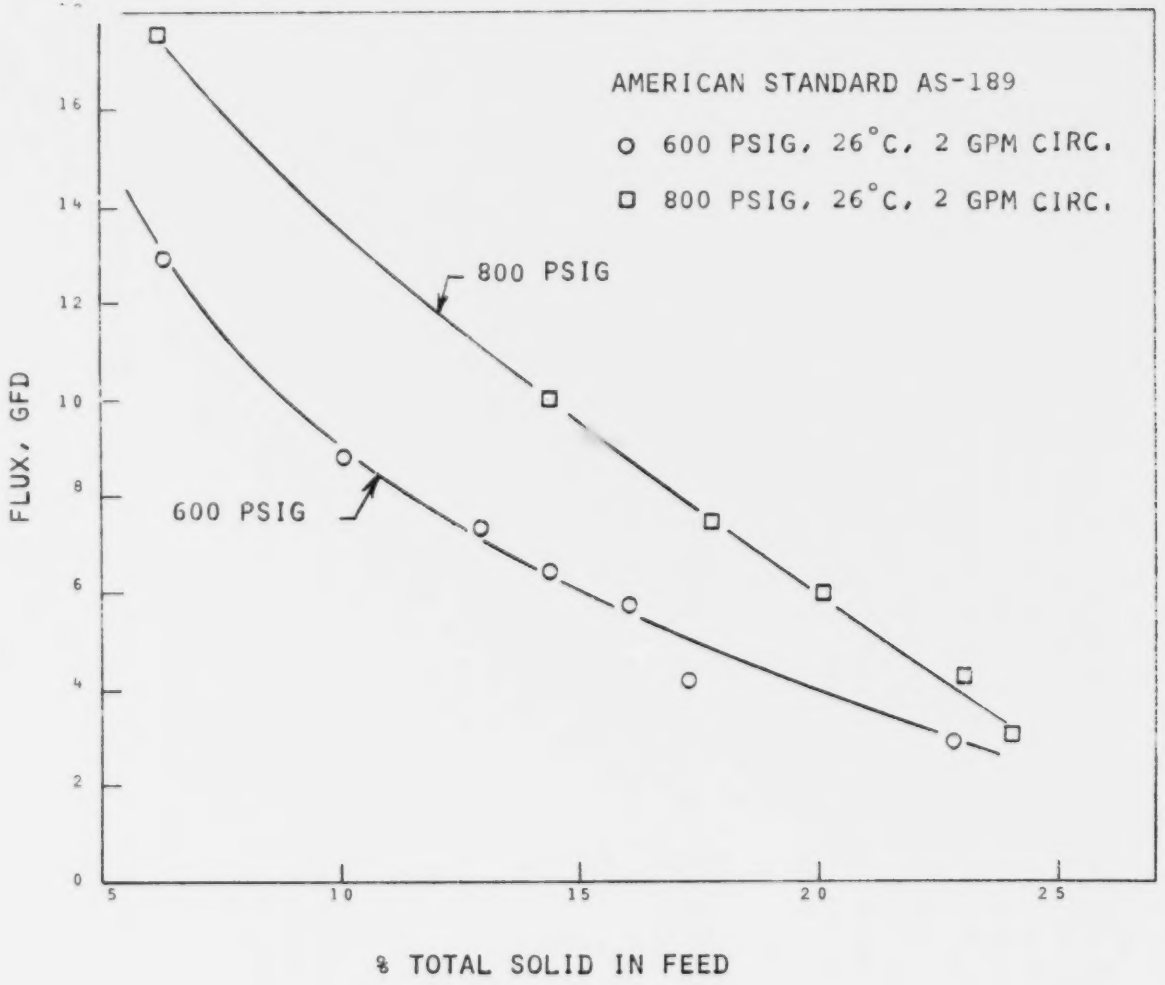


FIGURE 26

REVERSE OSMOSIS DATA: DEPENDENCE OF FLUX ON CONCENTRATION AND PRESSURE

Havens modules. The data obtained indicate that for operation in 3/8" to 1/2" tubes, a circulation rate of approximately 1.5 gpm is sufficient to eliminate the effects of concentration polarization. The circulation rate was chosen as a minimum for the pilot plant design.

Concentration of Whole Whey. Unfiltered cottage cheese whey was concentrated to approximately 24% solids in an experiment involving three different modules (American Standard AS-189, Havens 300 and Universal Water Corp.). Flux levels did not differ substantially from those observed with UF permeate. On this basis, it appears that the previous flux data can be used for design purposes for whole whey concentration. Similarly, the rejection data showed no major differences. This would be expected, since the only difference between whole whey and UF permeate is that the whey proteins have been removed from the latter.

Module Life. A close record was kept of module failures during prototype tests. This record is presented in Table 5. Shown are the name of the RO module, the date when the module was first tested, the date when module performance deteriorated, total hours of actual operation and the nature of the failure. Eleven Aerojet tubular membranes were tested. Although the rejection properties of the Aerojet tubes were the best, tube failure rate was unacceptable. Of the eleven tested, seven experienced rupture at an operating pressure of 500 to 600 psi. Similarly, Havens modules did not show the durability desired for the pilot plant. Of four tested, two failed after relatively short periods and two had unacceptably low rejection. Similar performance was observed with the Universal Water Corp. modules. Although these showed the highest flux and excellent COD rejection, failure of both modules occurred after a relatively short time. Three American Standard modules were tested, and all three performed well. One failure occurred due to a tube rupture at a point where a mechanical flaw existed.

It is difficult to determine the cause for module failures. One possibility relates to the fact that for several of the systems membranes were inserted inside tubular supports. Possibly upon startup or shutdown a partial vacuum could have existed within the modules causing membrane collapse. A second potential cause of failure involves micro-pulsations in pressure due to the use of a piston positive-displacement pump. Although an accumulator

TABLE 5: REVERSE OSMOSIS LIFE DATA; PERFORMANCE OF DIFFERENT RO UNITS

Name of RO Unit	Date (1)	Date (2)	Total hrs. actual use (3)	Nature of Failure
Aerojet Tubular Membranes				
#9B-290 (1.5ft^2)	2/10/70	3/25/70	10	End Seal Broke
9B-713 "	2/11/70	4/2/70	62	Tube ruptured in two places
0B-1105 "	2/11/70	4/6/70	136	Tube ruptured in one place
9B-581 "	2/10/70	4/9/70	106	Tube ruptured in one place
9B-792 "	2/11/70	4/10/70	166	Tube ruptured in one place
9B-633 "	3/26/70		160	Was good on last test date (4/22/70)
9B-595 "	4/7/70		36	Was good on last test date (4/22/70)
9B-813 "	4/9/70		30	Was good on last test date (4/22/70)
Aerojet New RO Tubes				
0B-3031 (1.5 ft^2)	8/6/70	9/22/70	228	Tube ruptured in one place
0B-2036 "	8/6/70	9/25/70	232	Tube ruptured in one place
0B-2033	9/24/70		4	Was good on last test date (9/25/70)
Havens Modules				
300 (17ft^2)	12/16/69		20	No gross failure before last test date (1/16/70), but had unsatisfactory rejection
210 "	1/30/70		8	"

TABLE 5 (Cont.)

Name of RO Unit	Date (1)	Date (2)	Total Hours Actual Use (3)	Nature of Failure
510 (17ft^2)	9/25/70	10/2/70	80	High flux and very low rejection indicating leak
610 "	8/6/70	8/15/70	15	"
Universal Water Corporation Modules				
395 (7ft^2)	12/29/69	1/14/70	14	"
349 "	1/30/70	2/2/70	8	"
American Standard Modules				
TM2-4 (1ft^2)	11/15/69		98	Was good on last test date 1/14/70
TM5-14 (AS-194) (9.5ft^2)	2/4/70		320	Was good on last test date 9/25/70
TM5-14 (AS-197) (9.5ft^2)	2/4/70	4/22/70	170	One tube ruptured due to mechanical flaw

1. When the RO unit was first tested.
2. When the unit performance drastically deteriorated
3. Includes time of runs with UF permeate and cottage cheese whey.

effectively damped out gross pulsations, micro-pulsations undoubtedly existed. These could have contributed especially to tube rupture.

On the basis of these limited performance data, a decision was made to use American Standard modules in the high pressure section of the pilot plant; that is, considerations pertaining to module durability were considered most important.

SECTION V

DESCRIPTION OF PILOT PLANT

Following prototype experiments, a two-stage pilot plant was designed, engineered and built by Abcor, Inc., and shipped to Binghamton, New York, for a four-month period of operation. This unit has since been shipped to and put in daily operation at Crowley's Milk Co. LeFargeville facility.

ULTRAFILTRATION SECTION (LOW PRESSURE)

General:

The UF section of the pilot plant contained 210 ft^2 membrane area (192 Abcor HFA-180 tubular membranes). These were arranged in four parallel passes (modules) contained in a stainless steel cabinet, fitted with sprays for cleaning and sanitizing purposes. At a typical flux of 13.5 gfd approximately 1,000 lbs of cottage cheese whey could be processed per hour.

Operation was batchwise; that is, 1 batch (or more) of whey was charged to the system per day and concentrated without adding additional whey. During operation, the feed was volumetrically concentrated 20-fold or higher, depending on the level of protein desired in the fluid concentrate. At the end of the run protein concentrate was recovered from the membrane unit by displacement with water or by draining.

Flow Schematic and System Components:

The UF section is shown in Figures 27 and 28, installed on site in Binghamton, N.Y. Shown in Figure 27 is the stainless steel sanitary cabinet, and in Figure 28, a closeup of the tubular membranes inside the cabinet. The system flow schematic is given in Figure 29. Whey from a large storage tank was fed gravimetrically to a smaller tank. The smaller tank held approximately 150 gals; the larger tank held about 4,000 gals, and was filled with fresh whey on a weekly basis. Whey was trucked from the Arkport, N.Y., cottage cheese production facility.

Whey was pumped from the small tank into the membrane circulation loop by a feed pump. High velocities over the membrane surface were maintained by a circulation pump, in order to maintain a high flux rate. The feed rate to the loop exceeded the flux and some of the loop material was bled back to the

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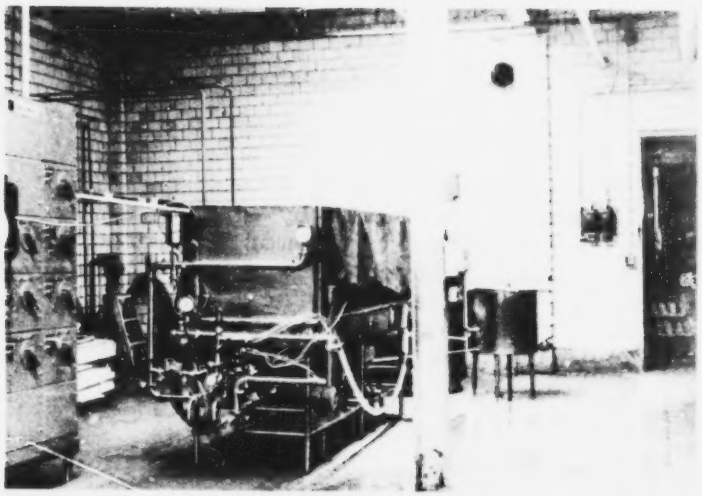

FIGURE 27

PILOT PLANT
SANITARY CABINET HOUSING ULTRAFILTRATION MEMBRANES

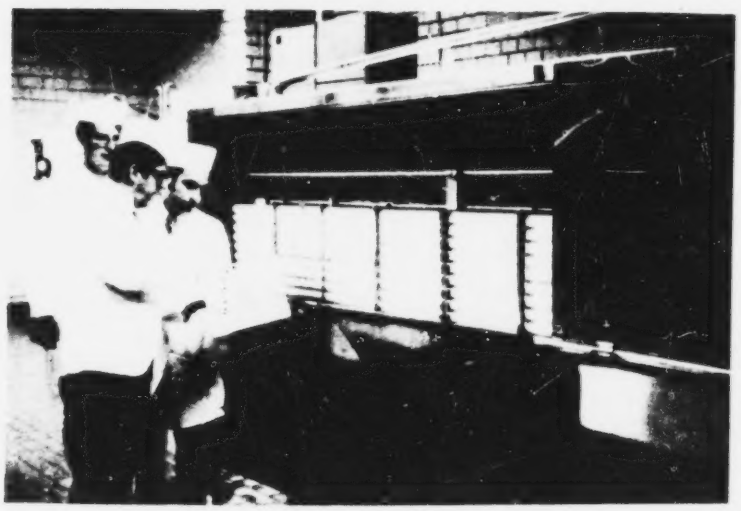

FIGURE 28

PILOT PLANT
ULTRAFILTRATION MEMBRANES IN SANITARY CABINET

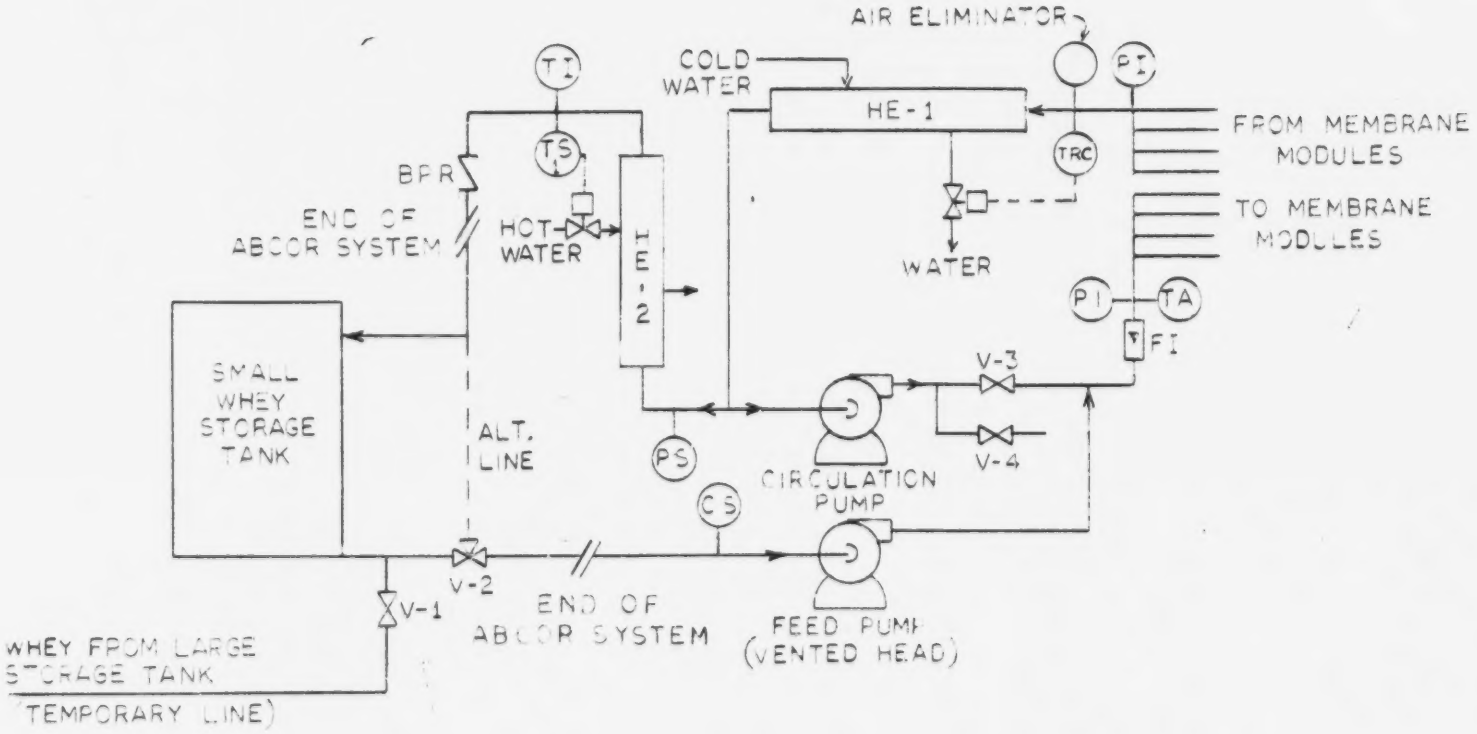

FIGURE 29 : ULTRAFILTRATION SECTION

small tank. This had the favorable effect of partially mixing the contents of the system, maximizing both flux and protein yield.

Two heat exchangers allowed the maintenance of a storage temperature higher than that in the membrane system. This minimized the growth of microorganisms since the bulk of the whey was held at a moderately high temperature during a run.

The components of the UF system and their function are described in Table 6.

A flow schematic for the clean-in-place system provided with the pilot plant is shown in Figure 30. This system was used to mix and circulate cleaning and sanitizing solutions through the membrane unit and also through the cabinet and module sprays.

REVERSE OSMOSIS SECTION (HIGH PRESSURE)

General:

The RO section of the pilot plant contained three stages. Each stage was operated with its own pump and control devices. The membrane areas of each stage were 90, 60, and 60 ft^2, for stages 1, 2, and 3 respectively. Stage 1 had 18 American Standard TM5-8 membrane modules; and stages 2 and 3 each had 12 modules. All contained RO-97 (AS-197) membranes. The modules were mounted in an open rack (Figure 31) for convenient cleaning of the exterior surfaces.

The purpose of the high pressure RO section was 2-fold: first, to concentrate the lactose permeate from the UF section, while reducing the effluent BOD; second, to concentrate whole whey, again with BOD reduction.

There were two modes of operation for the high pressure system. First, stage 1 was operated alone. With the interstage tank, UF permeate or whole whey was processed batchwise. Alternatively, stages 1, 2, and 3 were operated simultaneously in continuous operation. Feed was taken from the interstage tank and pumped through the 3 stage system, continuously removing both a fluid concentrate and low BOD permeate.

Flow Schematic/System Components:

A flow schematic showing all major system components is presented in Figure 32. A description of each item and its

function are given in Table 7. Figures 33 and 34 are photographs of the high pressure system. The first photograph shows the three control panels for the three high pressure stages, and the second shows the reverse side of the control panels.

The interstage tank is shown in Figure 27, just to the right of the UF membrane cabinet. In addition to accumulating permeate from the UF section for use as feed for the high pressure section, the interstage tank was also used to mix cleaning and/or sanitizing solutions for the high pressure system.

The electrical control panel for both the UF and RO sections is shown in Figure 35.

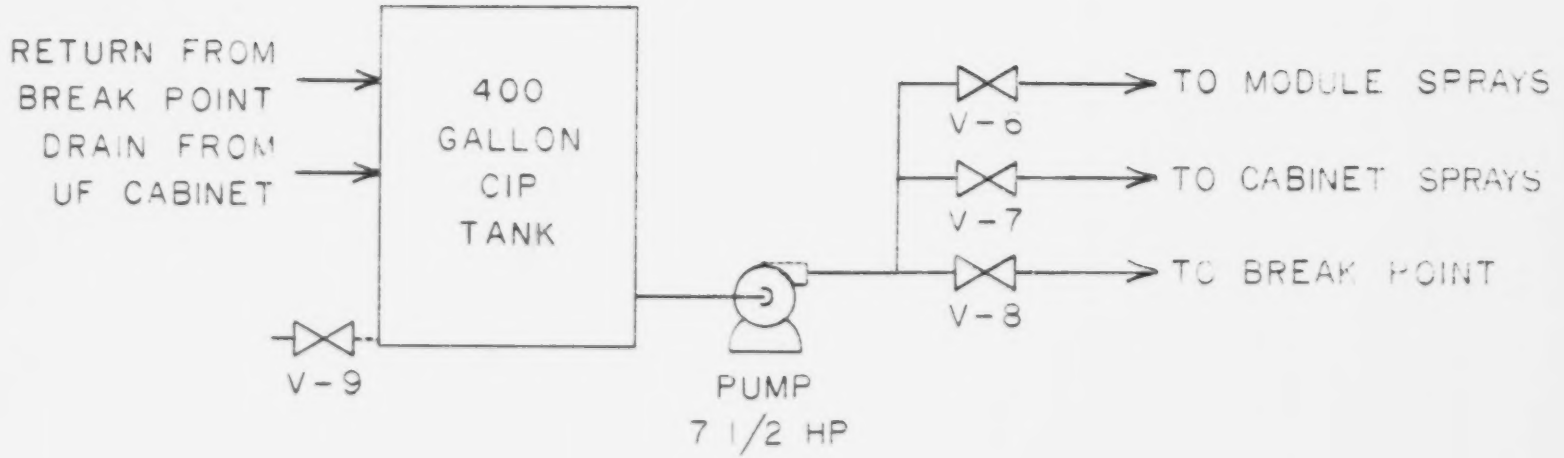

FIGURE 30: CIP SYSTEM

TABLE 6

ULTRAFILTRATION SECTION COMPONENTS

V1	Drain valve on whey storage tank.
V2	3-way plug valve.
V3	Diaphragm valve, for control of circulation rate in membrane system loop.
V4	Diaphragm valve, for draining system and sampling feed stream.
PBR	Back pressure regulator, for control of pressure on the low pressure side of the membrane modules.
CS	Conductivity switch, for shut down of feed and circulation pumps if air were entrained in whey feed.
FI	Flowmeter, for measure of circulation rate in membrane loop.
PI	Pressure guages.
TA	Audible temperature alarm.
HE-1, HE-2	Heat exchangers.
TRC	Temperature recorder/controller probe, connected to recorder for temperature measurement; also activated cold water solenoid valve on HE-1.
TI	Temperature indicating guage in return line to whey storage tank.
TS-1	Temperature switch, for control of hot water flow to HE-2.
PS	Low pressure switch, set at about 10 psi, for shut down of circulation pump at low system pressure.
Air Elim.	Air eliminator, for removal of air from whey in circulation loop.

TABLE 6 CONTINUED

ULTRAFILTRATION SECTION COMPONENTS

Feed Pump	Positive displacement pump with vented head set at 80 psi, for control of feed flow rate from whey storage tank to membrane system, and consequently - at a given flux rate through the membranes - the recycle rate through BPR back to storage.
Circ. Pump	Centrifugal pump, provided high circulation rate in membrane loop.
Alternate Line	1" pipe used for cleaning cycle.

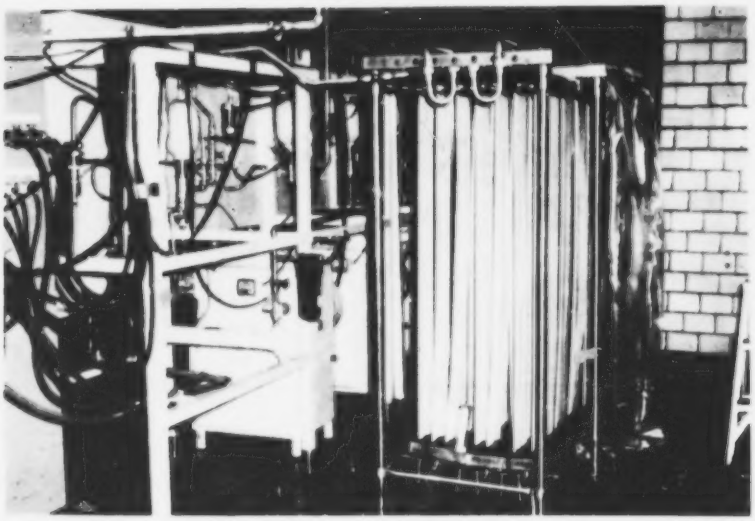

FIGURE 31

PILOT PLANT
REVERSE OSMOSIS SECTION MODULES

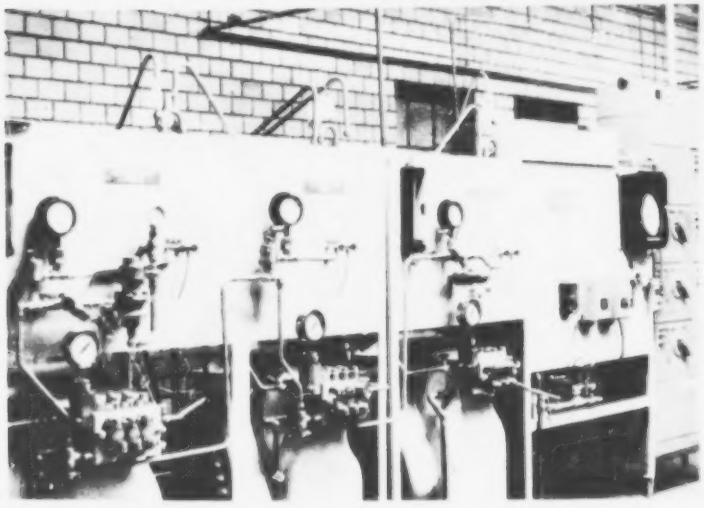

FIGURE 33

PILOT PLANT
REVERSE OSMOSIS SECTION CONTROL PANELS

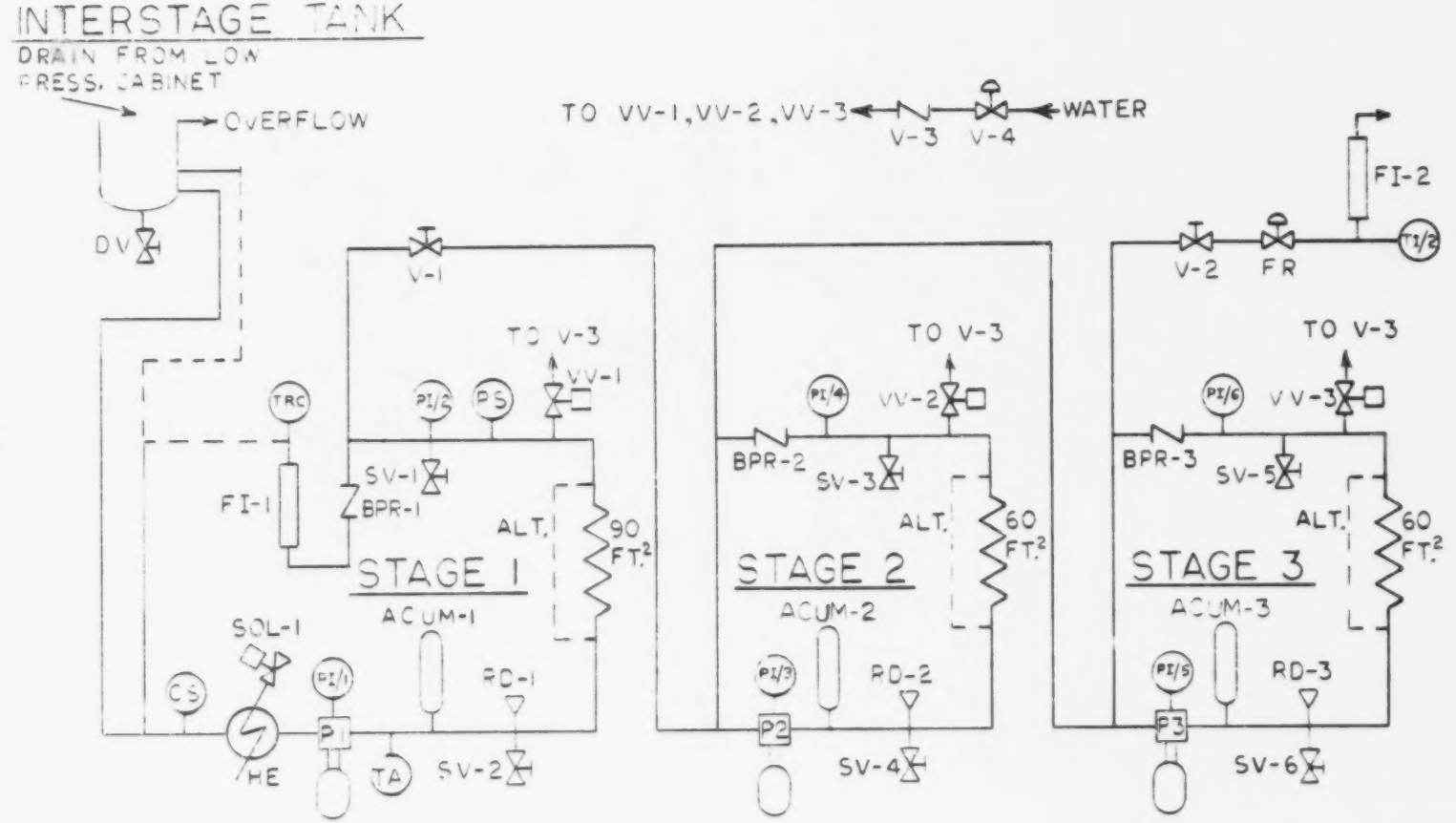

FIGURE 32: HIGH PRESSURE PIPING DIAGRAM

TABLE 7

REVERSE OSMOSIS SECTION COMPONENTS

P1, P2, P3	Homogenizers, used as high-pressure pumps.
ACUM/1, 2, 3	Pressure surge accumulators.
P1, P3, P5	Pressure guages on homogenizers' outlet ports, for measurement of pressure on high pressure side of membrane modules.
P2, P4, P6	Panel-mounted pressure guages, for measurement of pressure on low pressure side of membrane modules.
PS	Pressure switch, for shutdown of homogenizers if system pressure in stage 1 dropped below 60 psi.
CS	Conductivity switch, for shutdown of system if air were entrained in line to suction side of pump no. 1.
TRC	Temperature probe for recorder, actuated solenoid valve controlling cooling water flow to heat exchanger in stage 1.
HE	Heat exchanger, for lowering temperature of feed from interstage tank to stage 1 operating temperature.
SOL/1	Solenoid valve on heat exchanger, activated by TRC.
RD/1,2,3	Rupture disks, 1,000 psi rating.
SV's	Sample valves on inlet and outlet sides of membrane stages.
TA	Audible temperature alarm, indicated if feed from interstage tank were "too hot".
BPR/1	Stage 1 back pressure regulator, for control of operating pressure level in stage 1.
BPR/2,3	Stage 2 and stage 3 back pressure regulators, for increasing pressures of stages 2 and 3 differentially over that of stage 1.

TABLE 7 CONTINUED

REVERSE OSMOSIS SECTION COMPONENTS

FI/1	Flowmeter, indicated recirculation rate in stage 1.
V1	High pressure needle valve which could be used to isolate stage 1 from stages 2 and 3.
V2	High pressure needle valve, for shut-off of concentrate flow.
FR	Flow controller, for control of flow rate of lactose concentrate.
TI/2	Temperature probe, indicated temperature of concentrate leaving stage 3.
FI/2	Flowmeter, indicated flow rate of concentrate from stage 3.
VV/1,2,3	Solenoid vent valves; when system was not operating, water was introduced through these valves into the membrane modules to keep them wet during shutdown.
V3	A check valve installed in the water line to prevent fluid backing up the water line.
V4	Water regulator, controlled water pressure to membranes during storage.

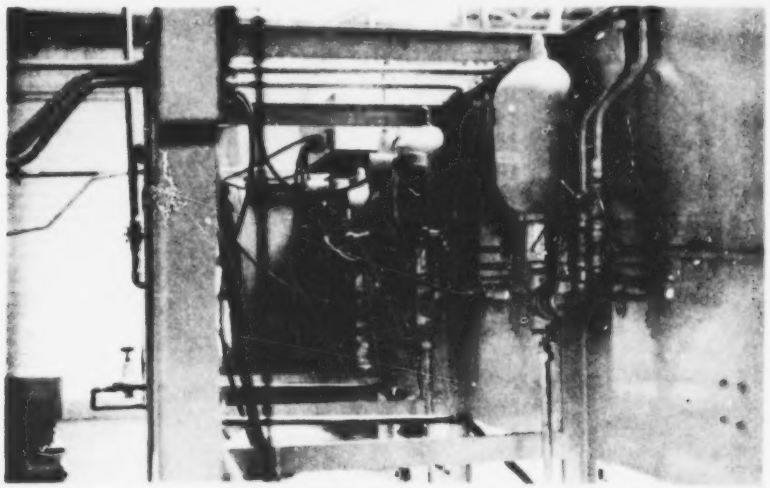

FIGURE 34: PILOT PLANT
REVERSE OSMOSIS SECTION CONTROL PANELS (REAR VIEW)

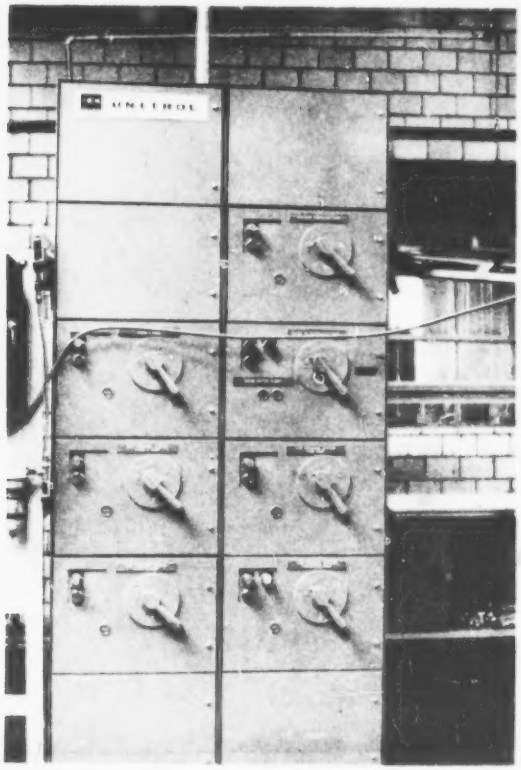

FIGURE 35: PILOT PLANT
ELECTRICAL CONTROL PANEL

SECTION VI

PILOT PLANT OPERATION

GENERAL PERFORMANCE

During the period from July 22 through December 1, 1970, the pilot plant was operated on a fairly regular basis. Operation was generally three days per week, with a fresh shipment of whey arriving each Monday. During this period pilot plant performance was excellent. Capacity remained unchanged with time, with the UF section having a capacity of 20,000 lbs whey/20 hour day, and the RO section having a capacity of 10,000 lbs whey/20 hour day. The BOD reduction observed in the bulk of the program was from approximately 35,000 mg/l to about 1000 mg/l (fresh, low-acid whey). Some exceptions were observed, in particular during periods when one or more of the RO high pressure modules developed leaks. In fact, the sole aspect of the pilot plant operation which was not entirely satisfactory related to the durability of the RO modules. At the beginning of the pilot plant operation several developed leaks and had to be replaced. In addition, over the program period four modules experienced tube ruptures. For the last two and a half months of operation, however, performance of the RO section was entirely satisfactory.

The system design proved to be sanitary and produced products with microbiological counts suitable as food or dairy products.

Details of the operation of the pilot plant are presented in the sections below.

SAMPLING AND ANALYTICAL PROCEDURES

Referring to Figures 29 and 32, the following Tables list sampling points:

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TABLE 8

SAMPLING POINTS FOR ULTRAFILTRATION SECTION

Number	Location	Purpose
1	Whey storage tank	For samples of raw whey taken at the beginning of a run
2	Return line to small whey storage tank (after BPR)	For samples of material in membrane circulation loop
3	Interstage tank (feed to high pressure tank)	For samples of UF permeate

TABLE 9

SAMPLING POINTS FOR REVERSE OSMOSIS SECTION

Number	Location	Purpose
1-6	SV/1 - SV/6	Sampling valves on inlet and outlet sides of high pressure modules - for samples of feed in each of the three circulating stages
7-9	High pressure permeate collection pans	For samples of permeate from each of the three stages
10	Flow through FI/2	For samples of final lactose concentrate

Analyses listed in Table 10 were performed on a regular basis.

ANALYSES USED FOR PILOT PLANT PROGRAM

Analysis	References
COD (Frequently calibrated with standard glucose solutions) BOD Kjeldahl Nitrogen Total Solids Acidity (Lactic Acid) Fat Conductivity	1. Standard Methods: Water and Wastewater, 12th Edition, 1965. 2. Standard Methods: Dairy Products, 12th Edition, 1965.

ULTRAFILTRATION SECTION

Procedure: All the experiments described involved unfiltered cottage cheese whey. The whey was stored warm until used, with the storage temperature ranging between 110 and 120°F. In one experiment a synthetic curd washwater was formulated by diluting one part of whey with four parts of cold water, and processed.

With this storage procedure operation was with whey with an acidity ranging between 0.5 and 1.3% (expressed as lactic acid). This is illustrated by the data of Figure 36. Whey was generally received with an acidity of about 0.5% (see Aug. 10 and 17, Sept. 7, Oct. 5 and 25, and Nov. 30). In the course of two to five days the whey acidity increased substantially. As will be shown in the discussion of the RO data, this increase in acidity led to an increase in the effluent BOD. This relates to the fact that lactic acid is only moderately retained by RO membranes.

Referring to Figure 29, operation in a batch mode was as follows. After a run the membrane system was cleaned according to procedures developed in prototype experiments. After cleaning, the system was flushed with

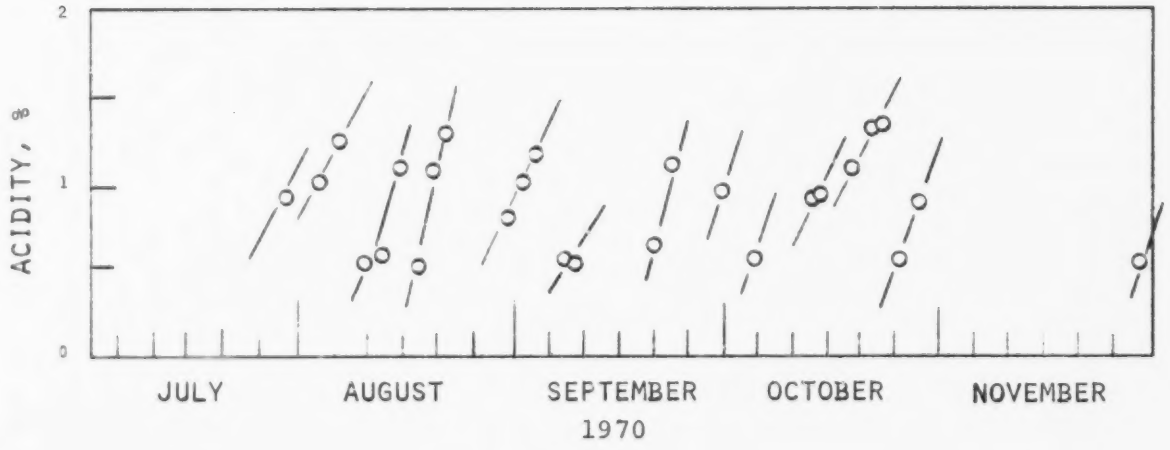


FIGURE 36

FEED WHEY ACIDITY

water and stored until the next experiment. Immediately before startup the system was sanitized with Antibac B.

Following flushing with whey, the small storage tank and the UF system were filled with raw whey. Upon startup whey was concentrated either for a given period of time or until a given volume of whey had been processed. During this period the small storage tank was kept full by the addition of fresh whey. At a pre-selected time or whey concentration the supply of fresh whey to the small storage tank was shut off. At this point the system contents were concentrated until the capacity of the small storage tank was exhausted. This latter operation is referred to as "cook-down".

Dependence of Flux on Protein Concentration: In Figure 37 are presented data for flux as a function of the retained protein concentration. The data presented are for runs on four days. The runs of October 19 and 22 were performed with whey of a high acidity. In addition, during the run of November 30 some acidification occurred in the whey at high protein concentrations. It appears on the basis of these data, as well as general observations on pilot plant performance (not discussed in detail here), that somewhat lower fluxes were observed with high acidity whey. Also shown in Figure 37 are the temperature profiles for the runs.

Flux decreased with increasing retained protein concentration, as expected. Data are comparable to prototype data which are also shown (from Figure 13 at 120°F and 13.5 gpm).

Dependence of Flux on Time (Life Data): In Figure 38 are shown flux data for the pilot plant during the 4½ months of operation. In the bottom plot is shown an average flux for batch operation, to an average concentration ratio (shown in middle plot), at an average temperature (shown in the upper plot).

The average concentration ratio is the concentration ratio up to the point of cook-down, and the average temperature is the temperature during this operation. In general, scatter in average flux up to cook-down can be related to variations in the concentration ratio and operating temperature. Higher fluxes are correlatable with lower average concentration ratios and higher operating temperatures. The most striking observation that can be made from the data of Figure 38 is that flux was virtually unchanged over the entire pilot plant operation. A mean average flux for a four-fold concentration ratio was 14 gfd at about 108°F.

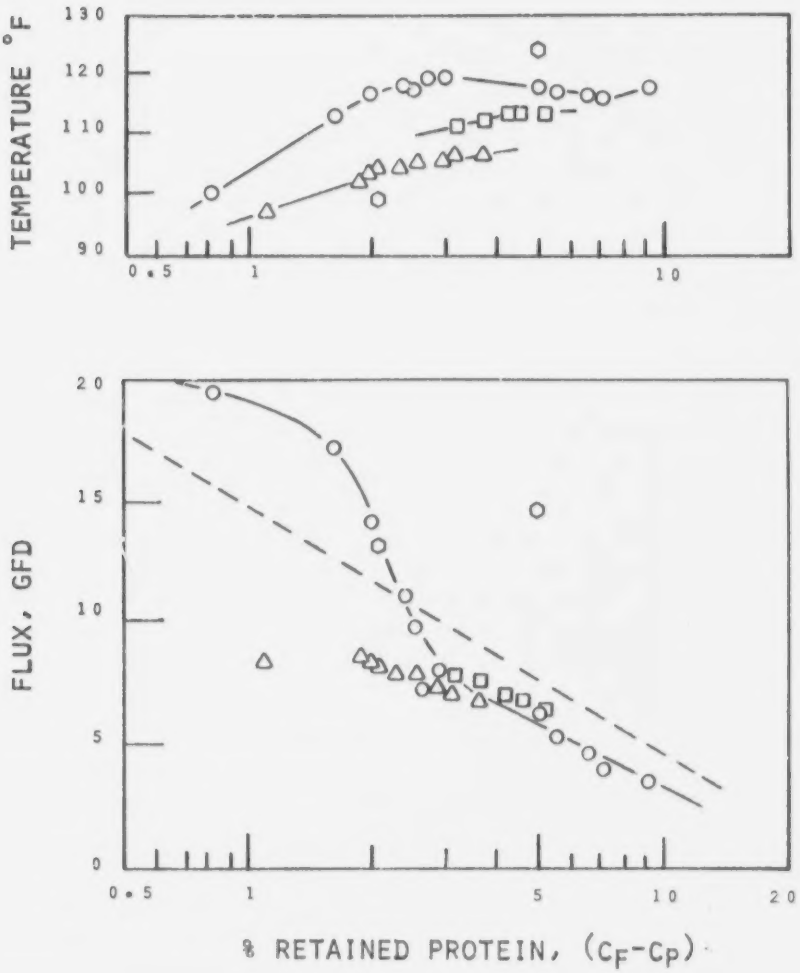


FIGURE 37
PILOT PLANT ULTRAFILTRATION DATA
EFFECT OF FEED CONCENTRATION ON FLUX

○ RUN OF 11/30/70
□ RUN OF 10/19/70
△ RUN OF 10/22/70
○ RUN OF 7/22/70

15.5 GPM RECIRCULATION RATE
INLET PRESSURE ≃ 60 PSIG
OUTLET PRESSURE ≃ 20 PSIG
---PROTOTYPE DATA (FIGURE 13)
(120° F, 13.5 GPM)

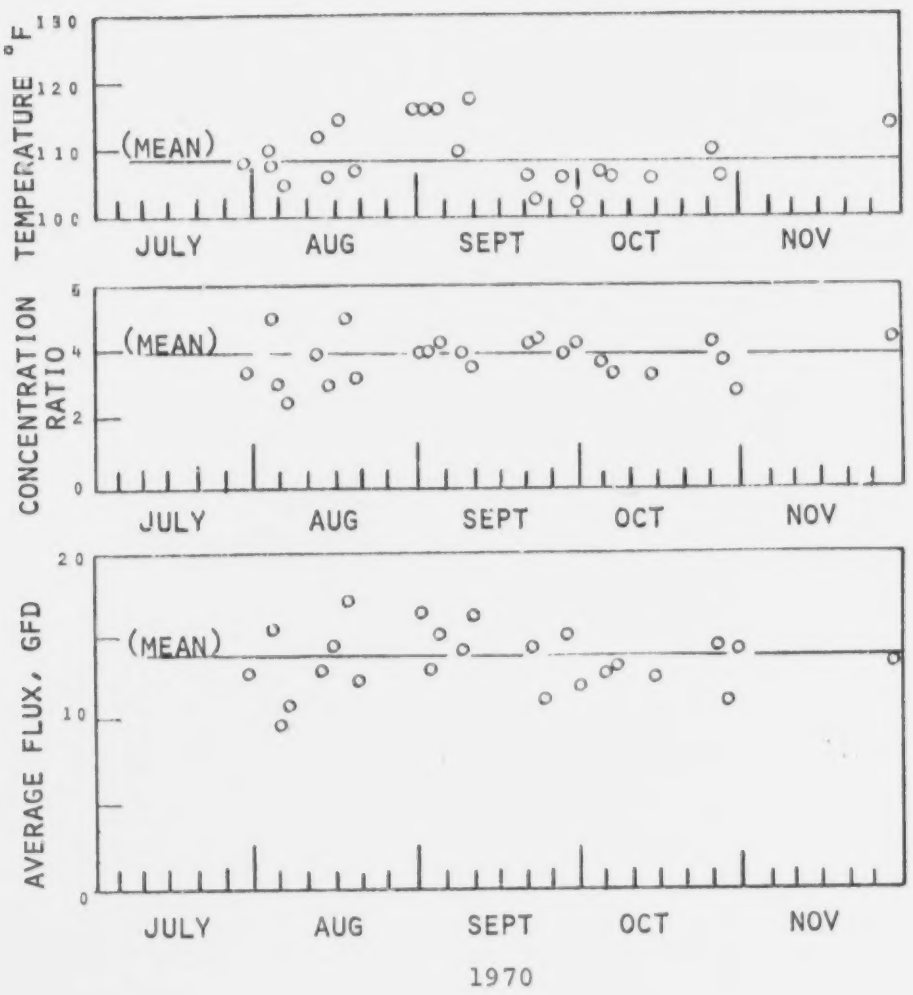


FIGURE 38

PILOT PLANT ULTRAFILTRATION DATA:
WHEY FLUX VS TIME

(15.5 GPM CIRCULATION RATE; INLET PRESSURE 60 PSI; OUTLET PRESSURE 20 PSI)

In Figure 39 similar data are shown for flux during the first and second hours of operation. Average flux during the first hour was 19 gfd and during the second hour about 14 gfd. These values were at an average temperature of 105°F. Although more scatter exists for these data, they confirm the conclusion that flux was unchanged during the pilot plant operation.

Additional flux data for the entire batch concentration, including cook-down, are shown in Table 11. Given are the date of operation, the protein concentration obtained in the final concentrate, the total solids content of the final concentrate, the percent protein on a dry solids basis, the average flux, and the operating temperature.

TABLE 11

PILOT PLANT ULTRAFILTRATION DATA

Date	Final Protein Concentration	Final Total Solids	Percent Protein (dry basis)	Avg. Flux (gfd)	Avg. Temp.
8/3	8.1%	14.6%	55.5%	14.4	110°F
7/29	6.4	12.9	50.0	10.7	100
8/17	14.6	21.1	69.5	13.7	114
8/31	8.3	15.5	53.5	14.5	112
11/30	9.3	15.8	59.0	13.0	114

Concentrates with protein percentages between 50 and 70% protein were generated, and average fluxes ranged between 10.7 and 14.5 gfd.

On the basis of the data for Figures 38 and 39 and Table 11, it is concluded that the capacity of the UF section of the pilot plant remained constant during pilot operation.

Dependence of Retention on Protein Concentration: At the end of the pilot program, protein retention was determined as a function of the protein concentration in the feed, in a manner identical to that discussed in "Prototype Experiments: Ultrafiltration". Protein retention data for the pilot plant are shown in Figure 40, along with prototype data from Figure 6.

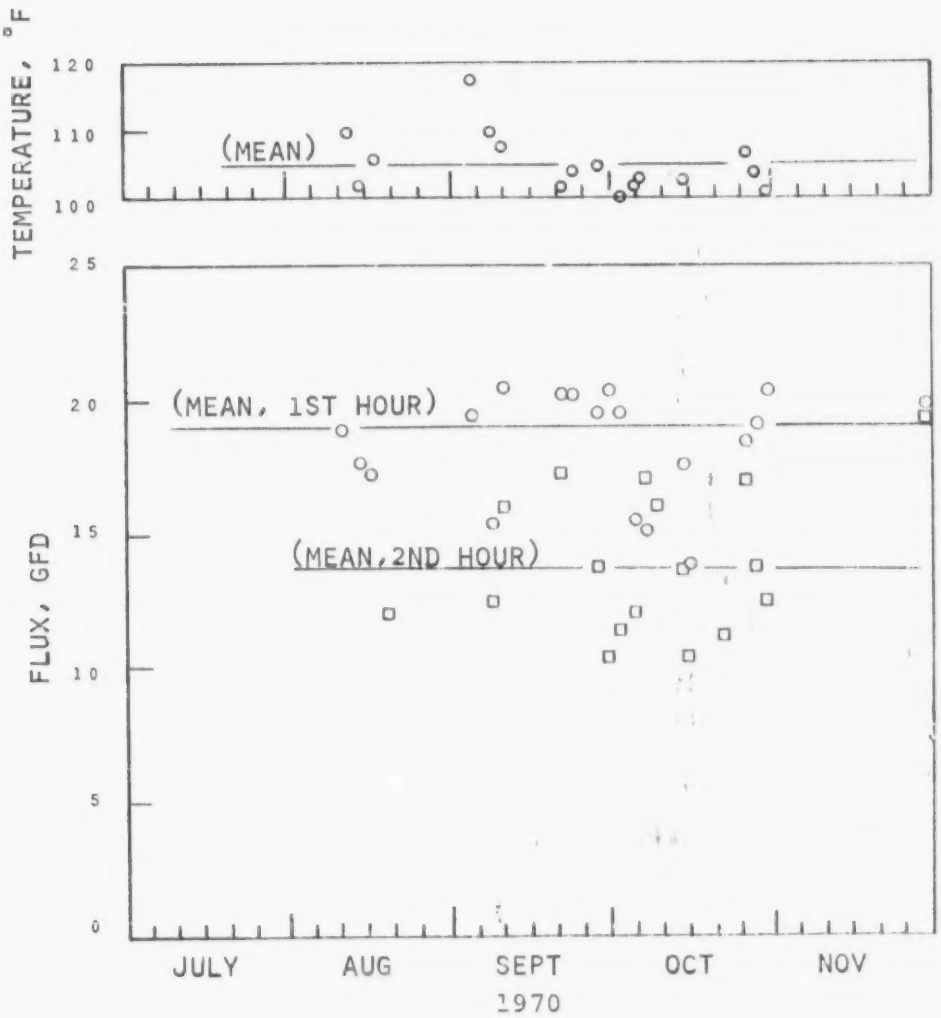


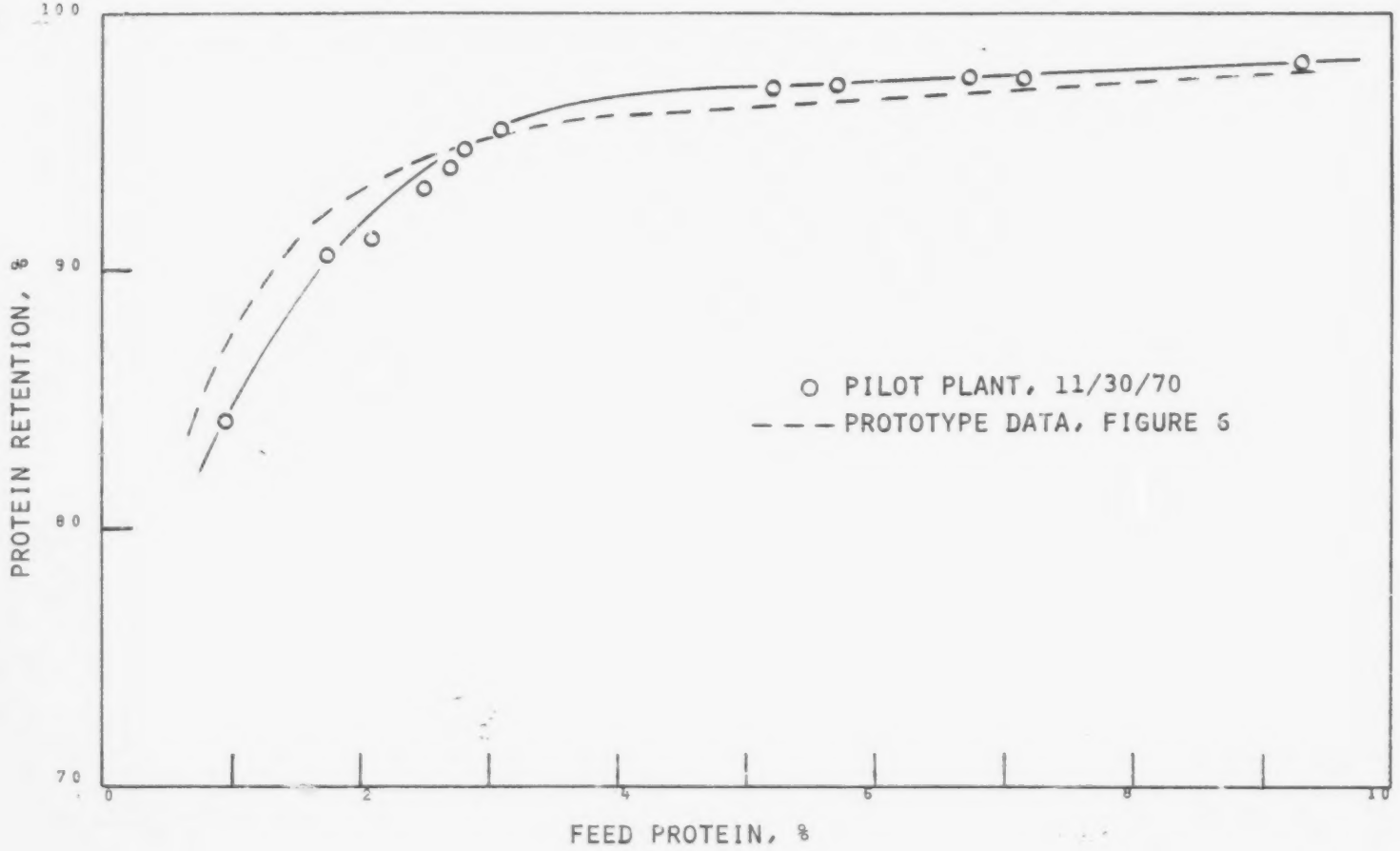
FIGURE 39

PILOT PLANT ULTRAFILTRATION DATA:
WHEY FLUX VS TIME

(15.5 GPM CIRCULATION RATE; INLET PRESSURE 60 PSI; OUTLET PRESSURE 20 PSI; ○ - 1ST HOUR; □ - 2ND HOUR)

FIGURE 40

PILOT PLANT ULTRAFILTRATION DATA: PROTEIN RETENTION



At low protein concentrations in the feed total protein retention was slightly lower with the pilot unit. This is hypothesized to be due to differences in the raw whey. At higher feed protein concentrations the pilot unit gave higher protein retentions. Retention at high protein concentration is more directly related to true protein retention properties of the membranes, and the pilot unit apparently had higher true-protein retention than the prototype membranes tested.

Since these data were obtained at the end of the pilot program and since no changes were made in the UF section during the program, it is concluded that protein retention was excellent throughout the pilot program.

Ultrafiltration Tests with Synthetic Curd Washwater: A synthetic curd washwater was formulated by mixing one part whey with four parts cold water. The temperature of the mixture was 74°F. This temperature approximates that of actual curd washwater. In a single experiment this mixture was concentrated 20-fold by volume (from approximately 0.12% to 2.5% protein) with an average flux of 14.5 gfd. The protein retention was excellent and followed the pattern exhibited for whole whey concentration in Figure 40.

Whey washwater can be concentrated at only slightly higher fluxes than whole whey, even though the protein concentration is 4 to 5-fold lower. Ultrafiltration flux is not substantially increased due to the fact that operation is at lower temperatures (70-74°F instead of 125°F).

This experiment demonstrated that curd washwater can be successfully concentrated, although to do so may not be economically attractive. A corresponding RO test for concentration of the permeate is described in a later section.

REVERSE OSMOSIS SECTION

Procedure: All RO experiments except one were performed with ultrafiltration permeate. The sole exception was an experiment involving whole whey concentration, described separately below.

Most membrane rejection analyses involved COD assays, which were correlated with BOD. Data for permeate samples from the RO section exhibited a relationship, BOD = ½ COD.

Toward the end of the pilot program it became apparent that the electrical conductivity of whey, UF permeates, and RO permeates could be correlated with COD. For RO permeates this is not surprising, since RO membranes have high salt rejections. Any decrease in the salt rejection of the mem-

brane would also be expected to lead to a decrease in the rejection of organic solutes. Data showing the overall conductivity-COD correlation are given in Figure 41. Over a wide range of COD levels a general relationship appears valid.

Operating procedure involved cleanup by flushing with line water until the "lactose-concentrate" stream was clear. The system was stored in this condition until the next run. Before startup the system was sanitized with Antibac B at a concentration of 1 oz/120 gal (10 ppm free chlorine), and purged with UF permeate.

Dependence of Flux and Rejection on Feed Concentration: Flux and COD rejection were determined as a function of feed concentration level. Data for a batch experiment (stage 1 only) are shown in Figure 42. Flux as a function of feed concentration, expressed as COD or approximate total solids, is shown in the upper curve. In the lower figure COD levels in the permeate are given; and in the middle graph, COD rejection.

These data show a decline in flux with increasing feed concentration in accordance with Equation 6. In addition, COD rejection was excellent, ranging from 98.5 to 99%. Based on these data the mixed permeate for a four-fold volumetric concentration would have a COD level of approximately 1250 mg/l. This would correspond to a BOD level below 700 mg/l.

In general, somewhat higher flux levels were observed during the pilot program. Figure 43 shows data for flux recorded during the period from July 22, through Oct. 21, 1970. Data points are shown for continuous operation with stages 1, 2 and 3 in three different temperature ranges. Numbers in parentheses near data points indicate approximate operating pressure. From stage 1 data, it is apparent that both increases in operating temperature and pressure resulted in an increase in flux. Similar observations are valid for stages 2 and 3. Data from Figure 42 from operation at 84°F and 700 psi are also shown.

The data have been scaled to an operating pressure of 750 psi in Figure 44. The scaled factor is based solely on the ratio of operating pressure; that is, fluxes are multipled by (750/operating pressure). This is a conservative scaled factor since it neglects the osmotic pressure of the feed, which is an important consideration especially for stages 2 and 3.

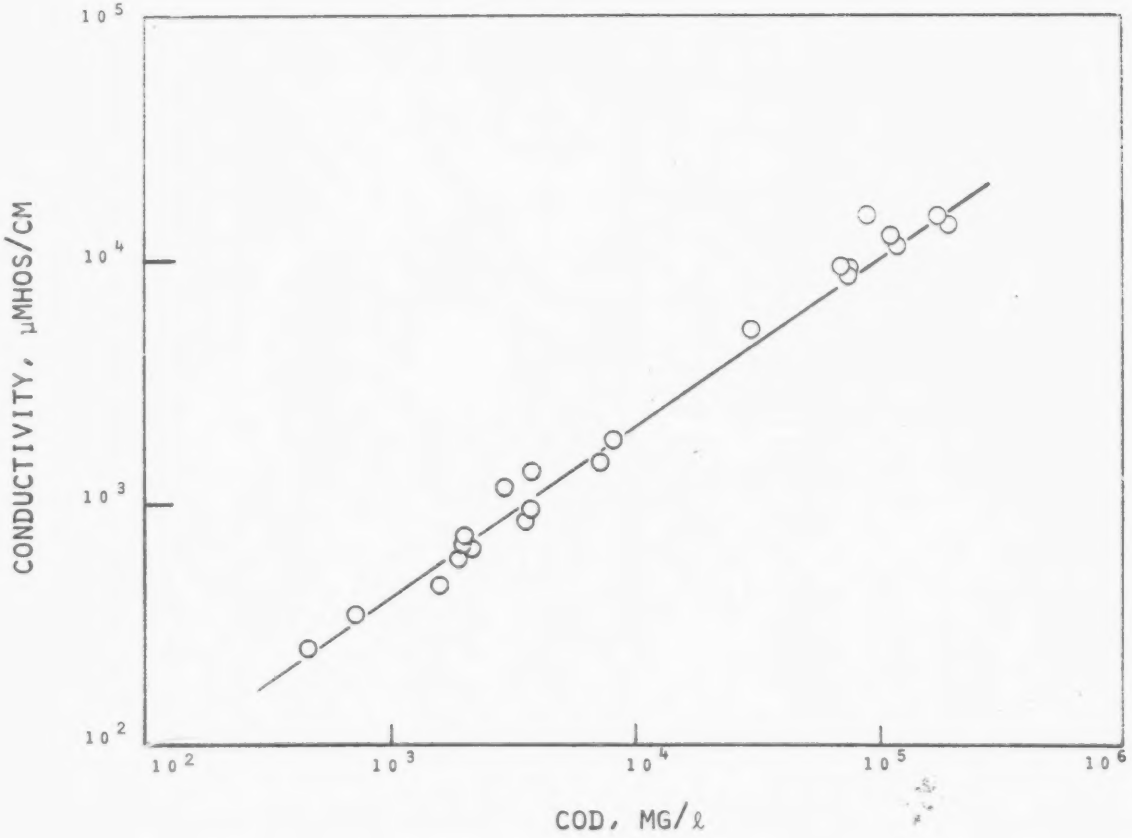


FIGURE 41

COD - CONDUCTIVITY CORRELATION FOR FEED AND PERMEATE OF REVERSE OSMOSIS SYSTEM

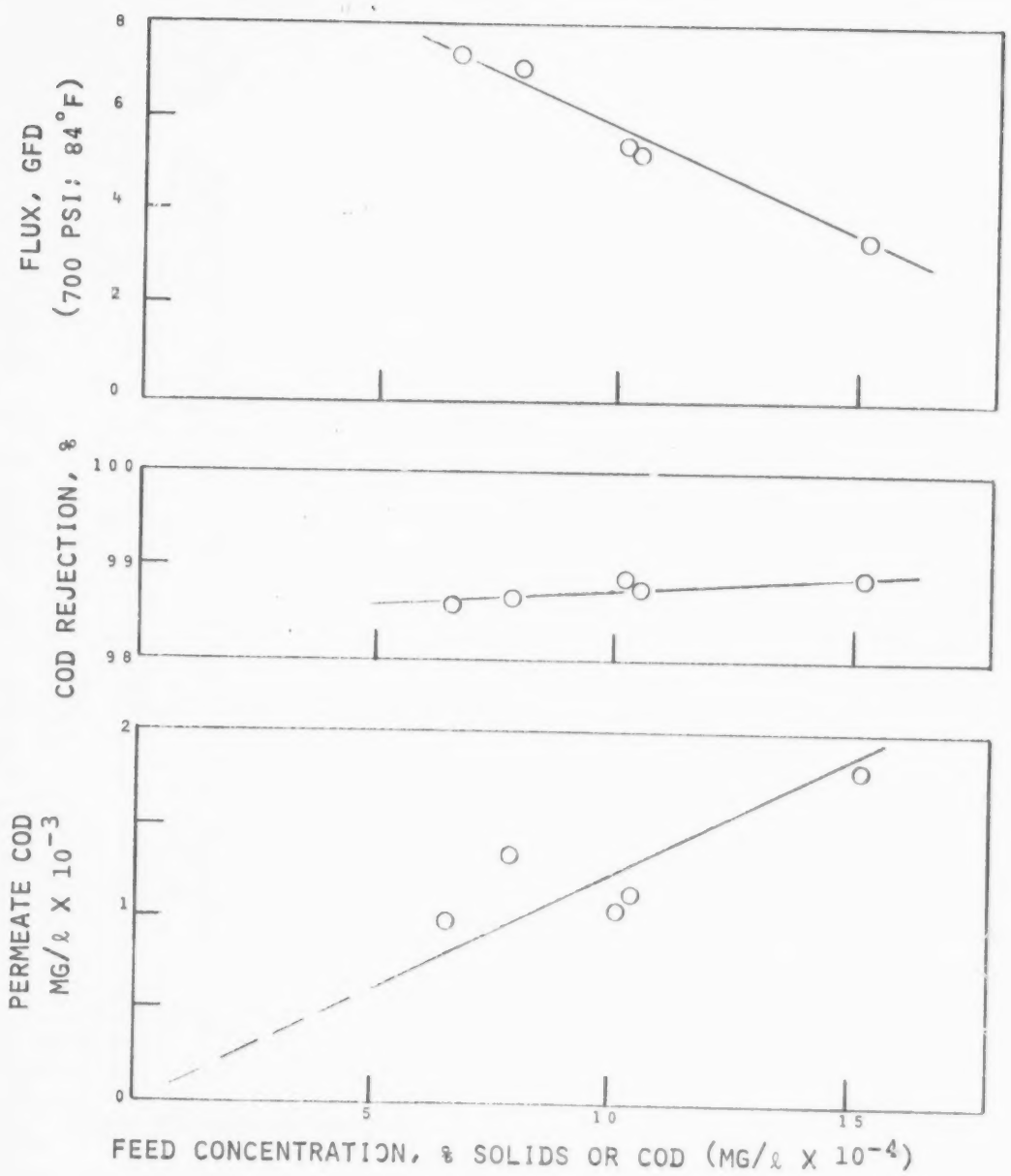


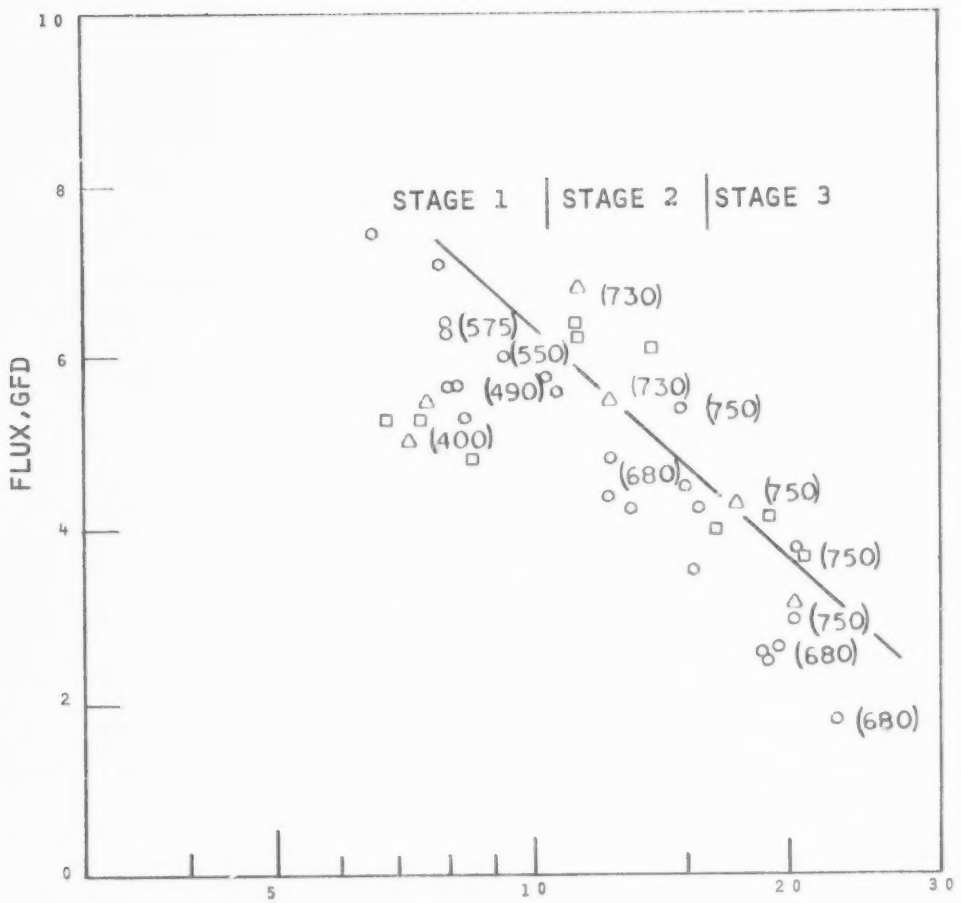
FIGURE 42

PILOT PLANT REVERSE OSMOSIS DATA

(8/31/70; WHEY ACIDITY = 0.8%)

FIGURE 43

PILOT PLANT REVERSE OSMOSIS DATA:
DEPENDENCE OF FLUX ON CONCENTRATION



FEED CONCENTRATION, % SOLIDS OR COD (MG/ℓ X 10^{-4})

○ FIGURE 42; 84°F; 700 PSI
○ 88 - 89°F
△ 82 - 84°F
□ 78 - 80°F

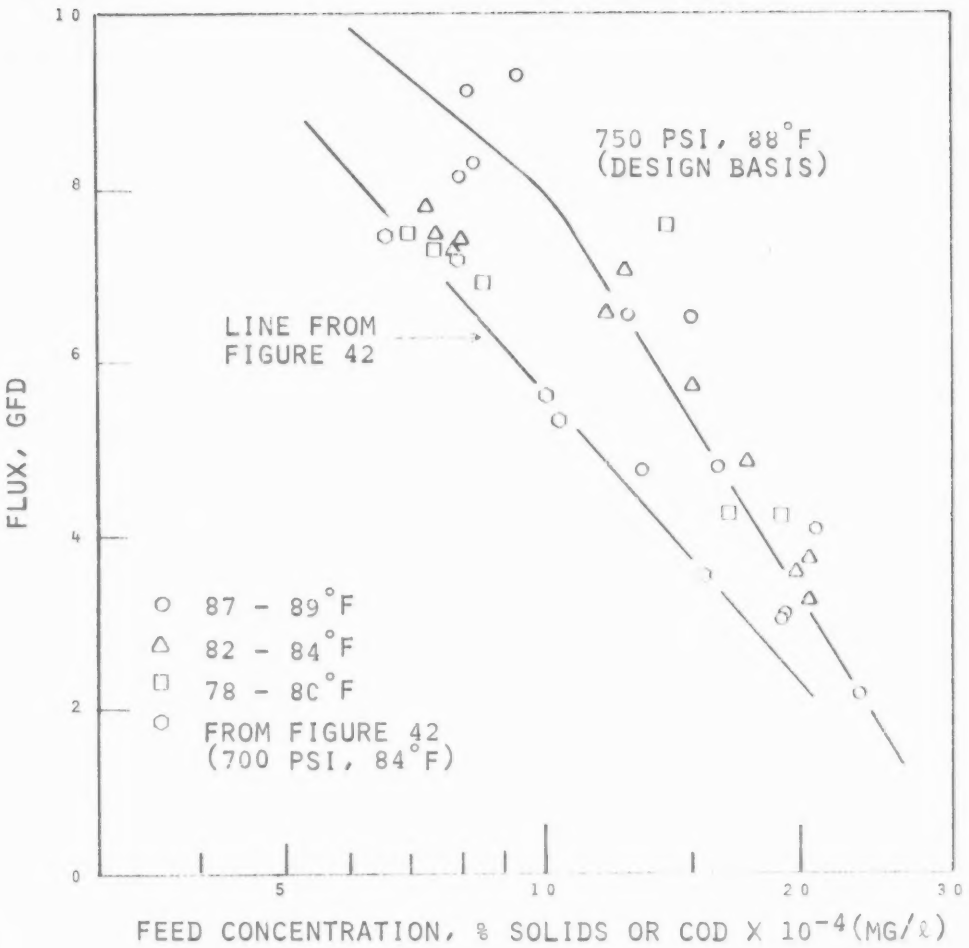


FIGURE 44

PILOT PLANT REVERSE OSMOSIS DATA:
DEPENDENCE OF FLUX ON FEED CONCENTRATION

(DATA FROM 7/22/70 THROUGH 10/30/70)

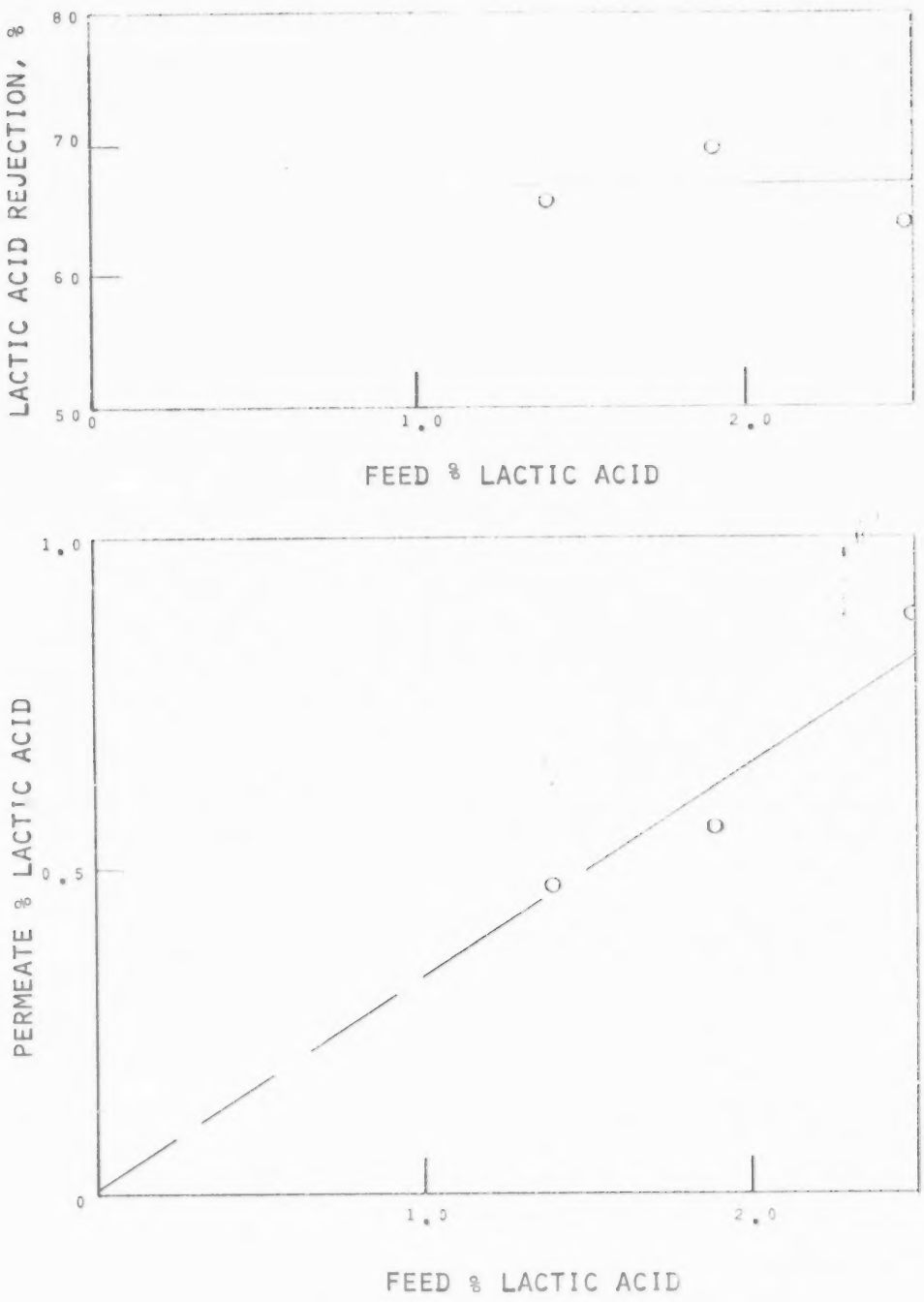


FIGURE 45

PILOT PLANT REVERSE OSMOSIS DATA:
LACTIC ACID REJECTION

(RUN OF AUG. 14, 1970)

No trend in the data could be related to length of operating time, and on this basis it is concluded that negligible membrane flux decline occurred.

Based on these data a conservative average flux for concentration of UF permeate from 6% to 21% solids is 5.85 gfd, and this value has been chosen as the design basis for the demonstration plant.

Lactic Acid Rejection: Pilot data showing lactic acid rejection are given in Figure 45. Shown in the lower plot is the level of lactic acid in the permeate as a function of the lactic acid in the feed. The three points correspond to samples from the three different membrane stages, operated in a continuous manner. From the upper plot, rejection is observed to be in the range of 60-70%. On the basis of these data and prototype data it is apparent that substantial lactic acid passes through RO membranes, and this contributes appreciably to the level of BOD in the final effluent.

Effect of Time on Flux (Life Data): In Figures 46, 47, and 48 are given water flux data for each of the three membrane stages. These data are for two different feeds: water and UF permeate. Flux values for the latter were measured after the system had been filled with whey, but before substantial concentration had occurred in stages 2 and 3. These fluxes were corrected for the osmotic pressure of the whey, assumed to be 100 psi. The correction was made according to Equation 6.

As indicated in these three plots, leaks were observed for all three stages on August 22. As will be demonstrated, these leaks were related to major decreases in membrane rejection. On September 1, the modules in the three stages were rearranged such that stage 1 contained only "on-spec" modules. The remaining modules were either returned to American Standard for new membranes (leaky modules) or were stored (good modules). On September 22, the remembraned modules were returned by American Standard and reinstalled in the pilot plant. Operation from this date proceeded smoothly, except that one module had a tube rupture and had to be replaced.

The modules in stage 1 were used throughout the pilot program. Life data on these modules, for the period through October 31, covers about 3½ months of operation.

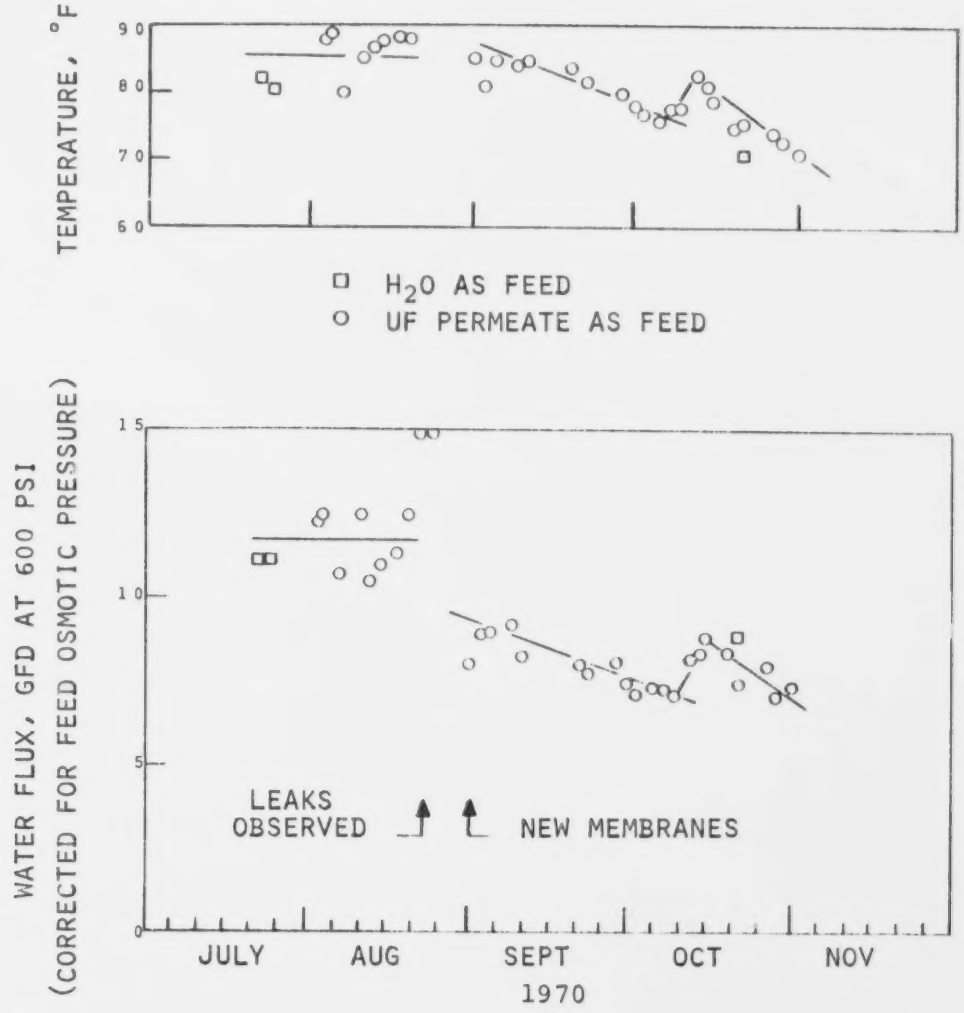


FIGURE 46

PILOT PLANT REVERSE OSMOSIS DATA: STAGE 1 FLUX

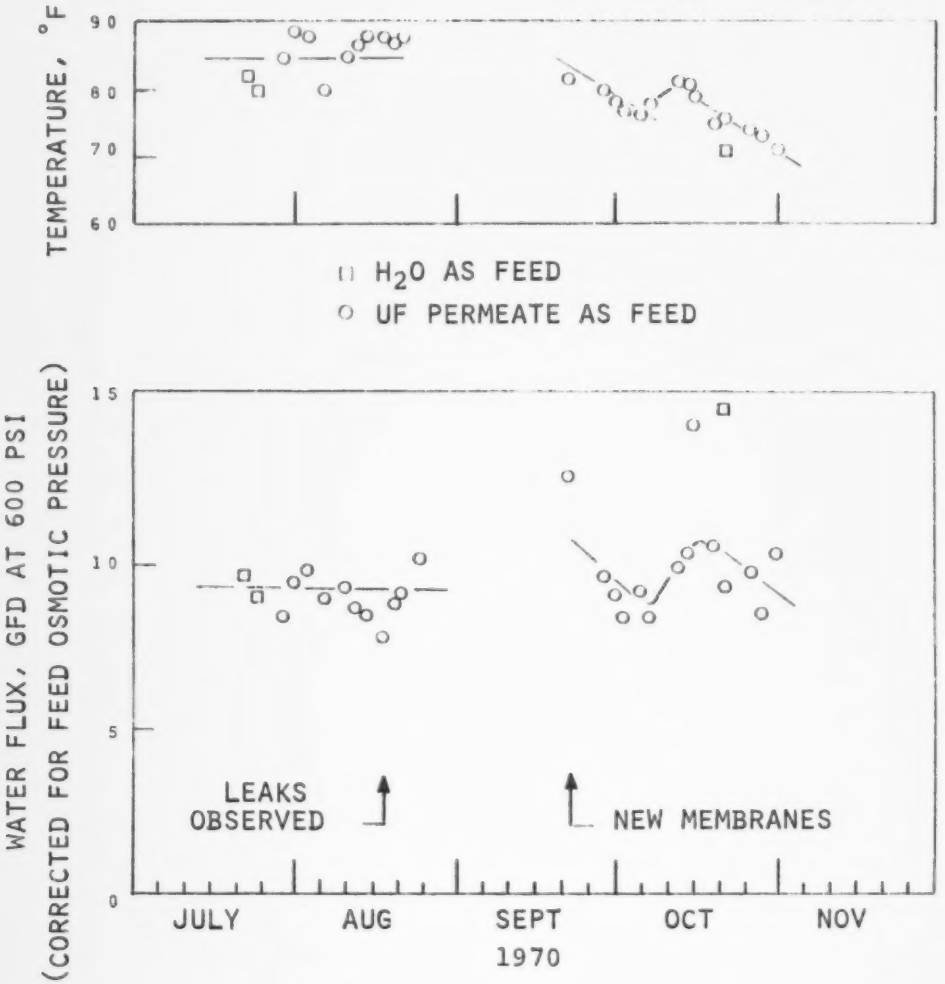


FIGURE 47

PILOT PLANT REVERSE OSMOSIS DATA:
STAGE 2 FLUX

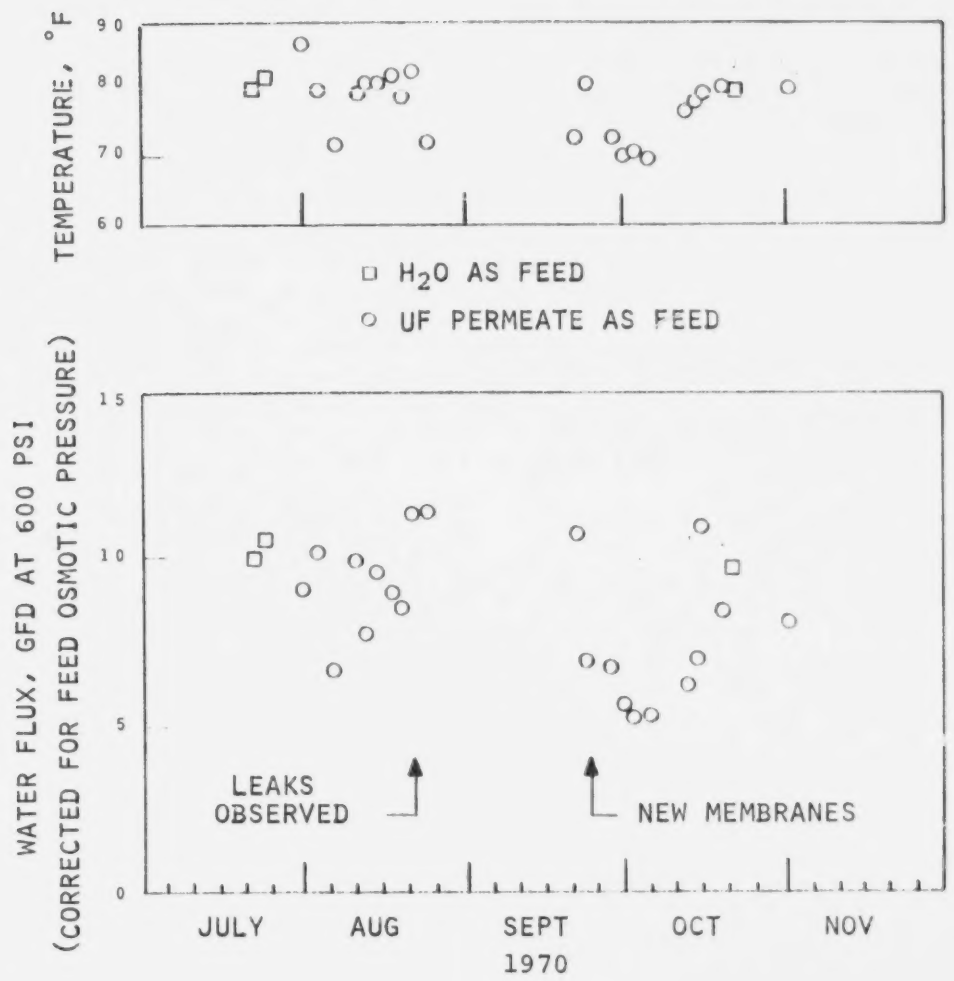


FIGURE 48

PILOT PLANT REVERSE OSMOSIS DATA:
STAGE 3 FLUX

As seen in Figures 46, 47, and 48, water flux at 600 psi for each of the stages was virtually unchanged with time, with decreases in flux during the months of September and October related to decreases in operating temperature. Water flux levels were between 7.5 and 10 gfd during periods of acceptable operation. The greater scatter for the data for stages 2 and 3 is due to inaccuracy in correcting solution flux for the osmotic pressure of partially-concentrated UF permeate.

Although flux data for the RO section show greater variability than data for the UF section, membrane flux for the RO modules remained relatively constant throughout the pilot program.

Effect of Time on Rejection (Life Data): Shown in Figures 49, 50, and 51 are rejection data for each of the three stages as a function of time. Operation began with high COD rejection for all three stages, followed shortly thereafter, by major deterioration in membrane rejection efficiency. This was related to visually observable module leaks (riboflavin appeared in the permeate, giving a green color). However, following the installation of new modules toward the end of September COD rejection became excellent. This performance continued through the end of November, at which time COD rejection for all three stages exceeded 97.5%.

All stages showed excellent COD rejection initially which dropped relatively quickly. This was related to the development of leaks in a few modules. This is demonstrated by the data of Table 12, which give rejection data on Sept. 10 for the stage 1 modules (see Figure 49). It is apparent that the bulk of the modules had acceptable to excellent rejection. One module had a gross leak; two had smaller leaks; and one was "off-spec." Similar decreases in COD rejection for the other stages were related to the development of leaks in individual modules.

Overall BOD and COD levels in the pilot plant effluent are shown in Figure 52. Also given are the operating pressure and feed whey acidity. On July 22, the first day of operation, the effluent COD was about 4000 mg/l, corresponding to a BOD of about 2000 mg/l. This was followed by a period during which many leaks occurred in the modules.

Continuous operation with all three stages began again toward the end of September. After this date, COD and BOD rejection were excellent, as shown. Variations in the oxygen demand of the effluent were related primarily to whey acidity. It is expected that operating pressure also played an important role, although these data do not indicate any marked effect. This conclusion is based, however, on the prototype data presented in Figures 20 through 22.

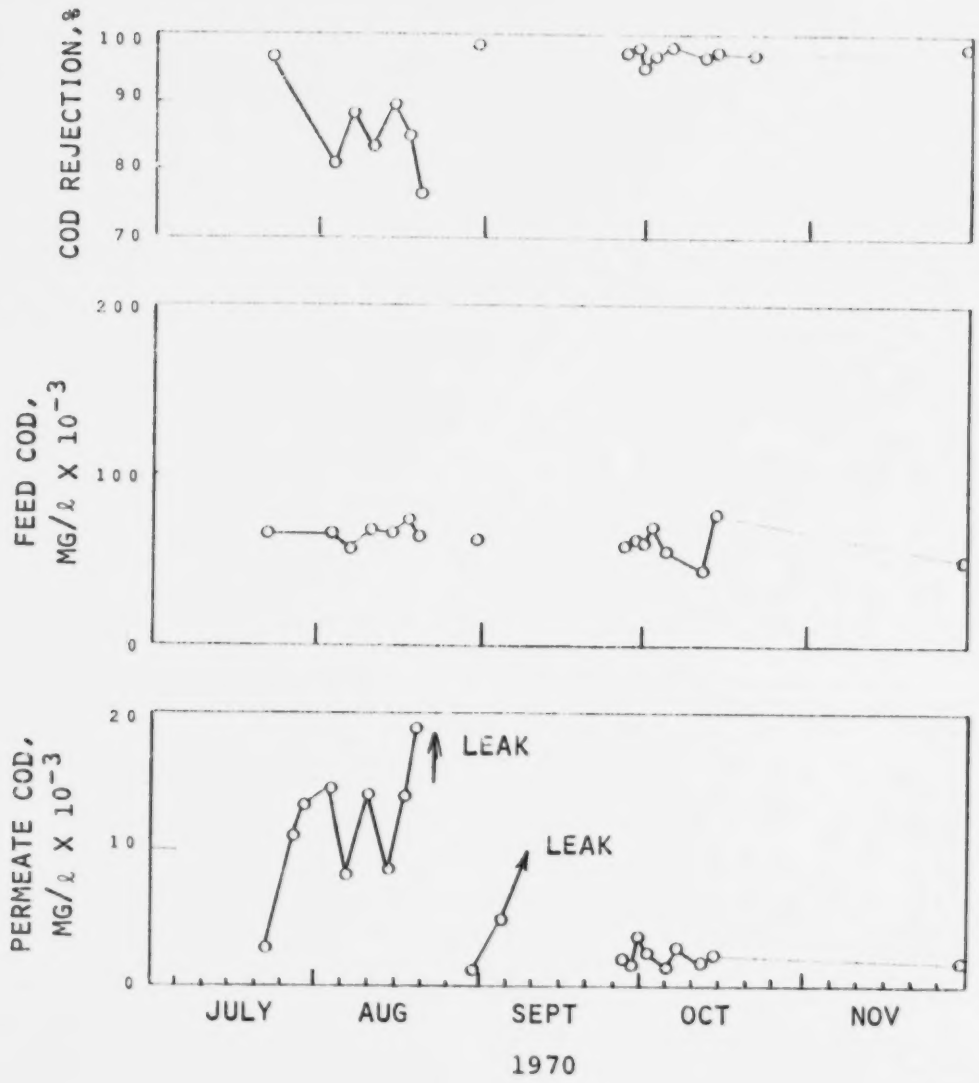


FIGURE 49

PILOT PLANT REVERSE OSMOSIS DATA:
STAGE 1 COD REJECTION

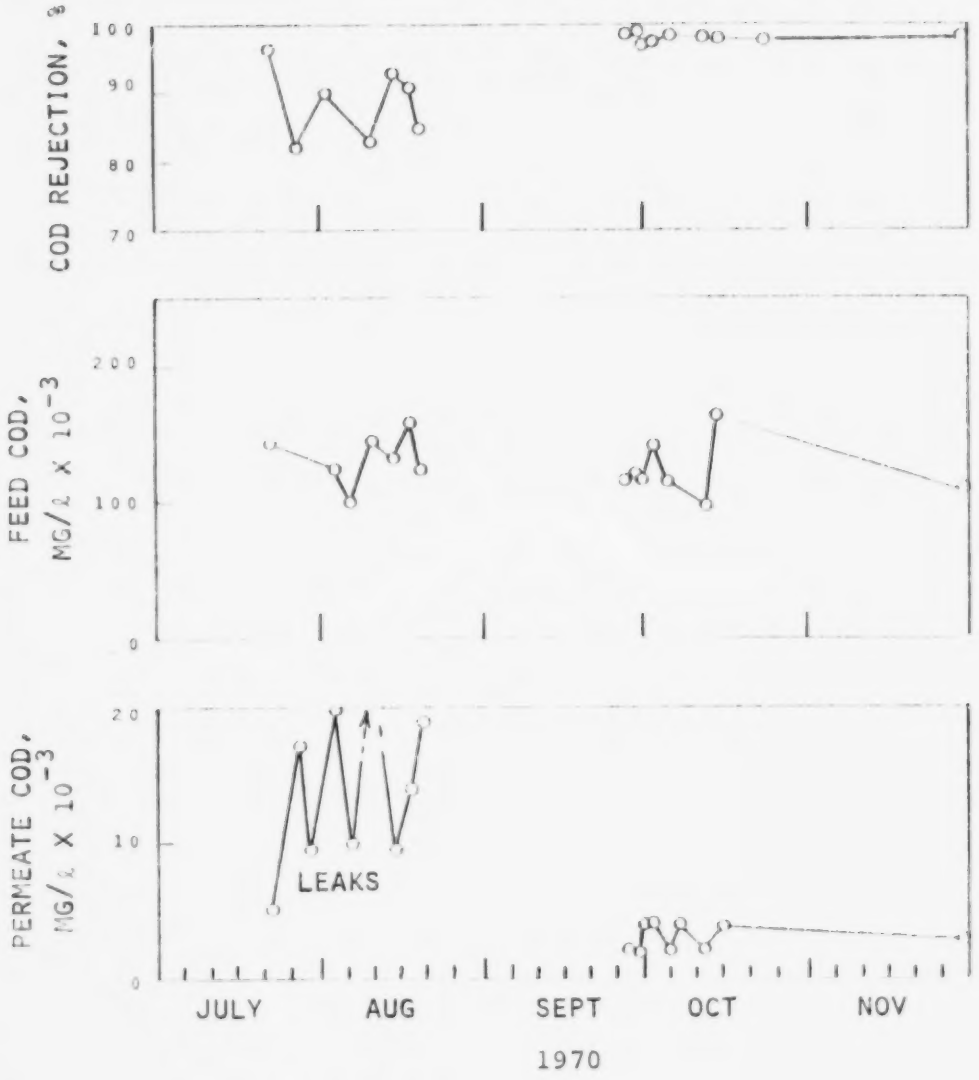


FIGURE 50

PILOT PLANT REVERSE OSMOSIS DATA:
STAGE 2 COD REJECTION

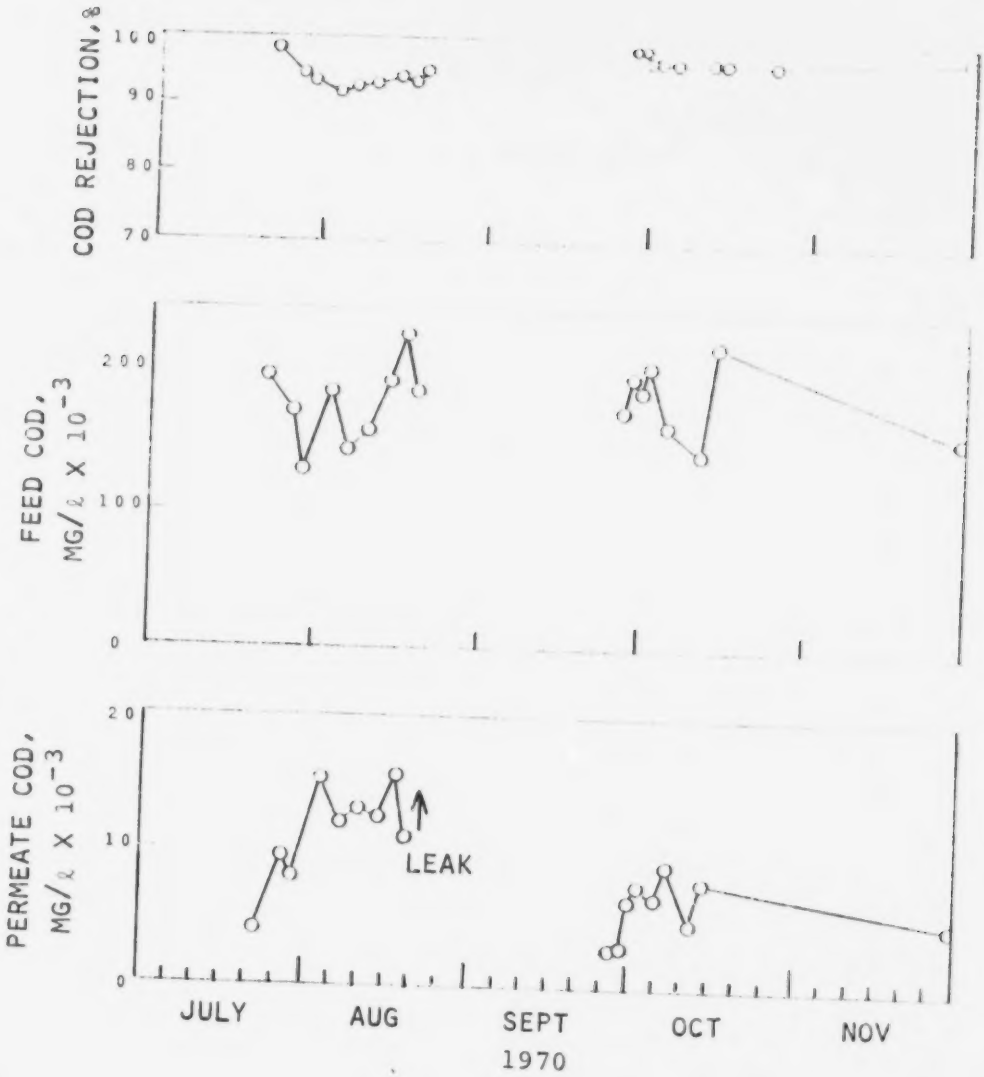


FIGURE 51

PILOT PLANT REVERSE OSMOSIS DATA:
STAGE 3 COD REJECTION

TABLE 12

COD REJECTION DISTRIBUTION OF MODULES IN STAGE 1
ON 9/10/70

Module #	COD Rejection, %	Observations
1	99.3	
2	99.2	
3	99.2	
4	99.0	good rejection
5	99.0	
6	98.9	
7	98.8	
8	98.7	
9	98.3	
10	98.3	
11	98.1	acceptable rejection
12	97.3	
13	97.3	
14	96.5	
15	91.6	"OFF. SPEC.", unacceptable
16	86	leak,
17	84	leak,
18	50	gross leak

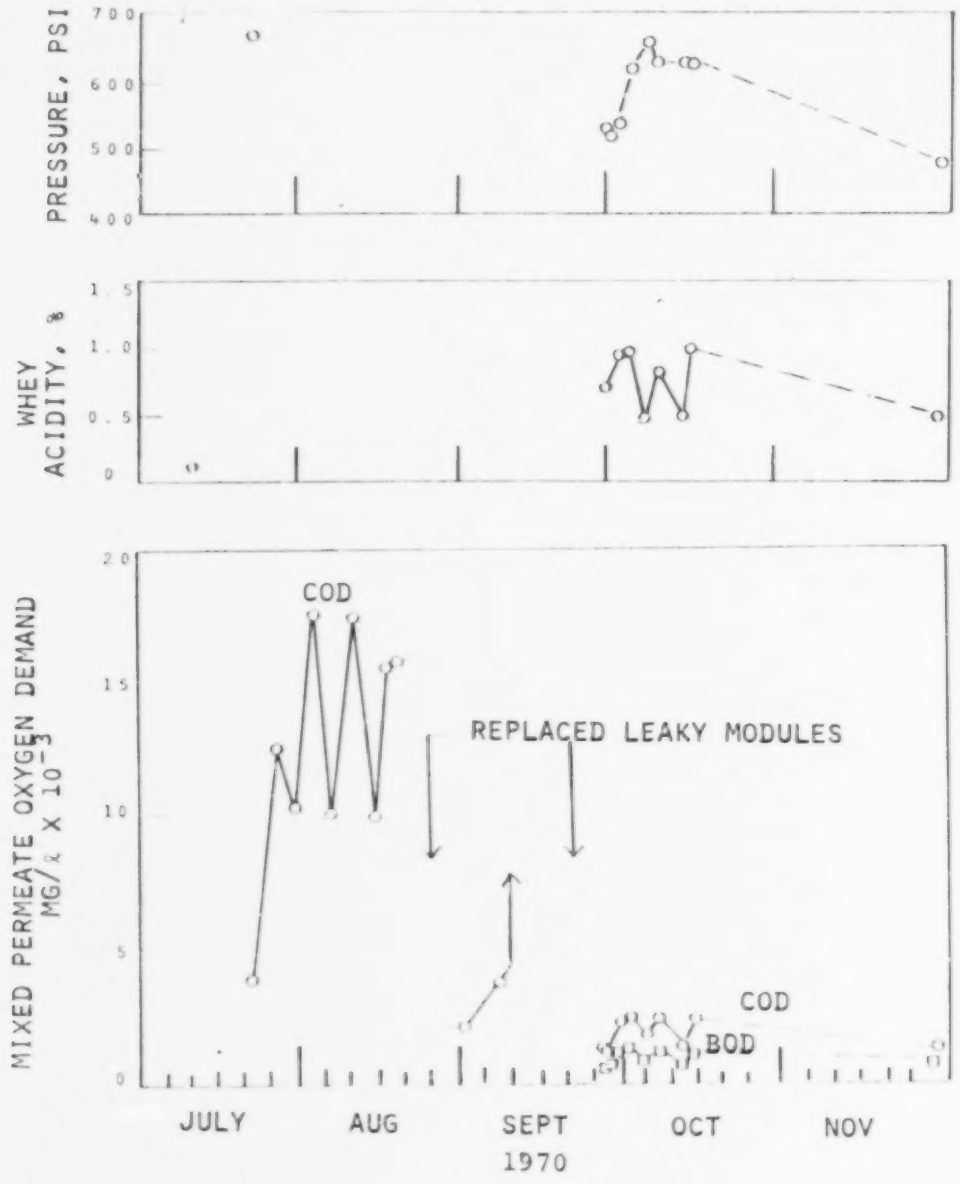


FIGURE 52

PILOT PLANT REVERSE OSMOSIS DATA:
OXYGEN DEMAND REDUCTION

(RAW WHEY COD ≈65,000 MG/ℓ; BOD ≈35,000 MG/ℓ)
(≈ 4-FOLD CONCENTRATION TO 22 - 25% SOLIDS)

BOD levels, on days when low-acidity whey was used, were in the range of 1000 to 1500 mg/l. This corresponds to an overall BOD reduction of 96-97% based on a raw whey BOD of 35,000 mg/l. Reduction for the demonstration plant is expected to be even greater since the demonstration plant will be a once-through design, as opposed to a three-stage circulation system. The latter design, used in the pilot plant, is less efficient for BOD reduction, because UF permeate is processed at higher concentrations than would be the case for a once-through system.

Miscellaneous Reverse Osmosis Tests. (Concentration of Whole Whey and Curd-Washwater UF Permeate): In a single experiment, raw cottage cheese whey was concentrated in the RO section. This test, performed on October 27, demonstrated that flux and rejection in the high pressure section for whole whey concentration were comparable to values obtained for the concentration of UF permeate. Similar results have been discussed above for prototype experiments. One marked difference between performance with whole whey and UF permeate related to the ease of system clean-up. As will be seen in the section on microbiological data, when UF permeate was processed in the RO section, clean-up and sanitizing was relatively simple and effective. However, in the experiment with whole whey some difficulty was encountered in clean-up. Probably the presence of residual casein fines as well as the whey proteins contributed to both system fouling and the accumulation of solids at system dead-ends. On the other hand, UF permeate is de-proteinized and contains no suspended solids. Lactose, salts and the residual low molecular weight organics have high solubilities and can be removed from the system simply by flushing with water. This difference between concentrating whole whey and UF permeate by RO appears to be quite important, in that some currently available RO equipment may be difficult to clean. Processing of UF permeate alleviates this process consideration.

When the synthetic curd washwater (20% whey in cold water) was processed by UF, the permeate was processed in the RO section. COD rejection in all three high-pressure stages exceeded 98%. Fluxes were observed to be higher than for the treatment of UF permeate from whole whey. This was as expected, since the permeate from the synthetic curd washwater had a 5-fold lower solids content. Concentrating the washwater UF-permeate 5-fold by volume produced a concentrate with approximately 5% solids. Flux for all three high-pressure stages was approximately 8 gfd at 72°F and 500 psi. The processing of washwater/UF-permeate by RO presents no obstacles, and as an approximation the membrane area requirement can be conservatively based on considering the washwater to be whole whey.

MICROBIOLOGICAL DATA

During the course of the pilot-plant program, samples were analyzed for total viable bacteria and coliforms. In Table 13 are given the data for different test dates. The dates run from the end of July through the beginning of October. In the second column are given the dilution ratios for the samples before culturing. The other columns give counts for raw whey, UF concentrate, UF permeate, RO feed to stage 1, RO feed to stage 2, RO feed to stage 3, final lactose concentrate, and the permeates from RO stages 1,2, and 3.

Several observations can be drawn. First, at no time did the counts for either total viable bacteria or coliforms in the protein concentrate exceed the level for grade A milk (50,000 org/ml). This was the case even though counts in the feed whey sometimes exceeded this level (see data for July 31, August 18, September 29, and October 5).

Evidently, operation in the UF section at 110-120°F for several hours did not lead to bacteriological growth. In fact, in certain cases it appeared that decreases in plate counts were observed. These data confirm the previously discussed prototype data which indicated that growth of microorganisms in cottage cheese whey is not a factor of major importance.

In general, the UF permeate, sampled in the interstage tank, also had low total plate counts. There were some exceptions, however, which are attributed to improper cleaning. Although the counts on August 14, September 29, and October 1 were high, proper cleaning of the system resulted in a return to low counts. (See for example, data of August 18 and October 5.)

The feed in RO stages 1, 2, and 3 was taken from the interstage tank. Correspondingly, on days when the UF permeate was contaminated (August 14, September 29, and October 1), the feed to the RO section was also contaminated. This explains the high counts on the same dates in the RO feed samples. Additional data for October 12 and October 28 showed contamination of the high pressure section even though the UF permeate had low counts. This is thought to be due to the fact that no sanitizer was used on those days.

In general, plate counts for the lactose concentrate followed the counts for the feed to the RO system. At times, however, counts in the lactose concentrate were higher than in the RO system (e.g., July 29), and this is thought to be due to the use of a Tygon tube to drain off the lactose concentrate.

TABLE 13

BACTERIAL DATA
TOTAL PLATE COUNTS AND E. COLI COUNTS

DATE	DILUTION	RAW WHEY		UF CONCENTRATE	
		TPC	E.COLI	TPC	E.COLI
7/29/70	1/1000 1/10,000	12,000 20,000	<1	20,000 30,000	<1
7/31/70	1/1000 1/10,000	41,000 111,000	<1	2,000 <10,000	<1
8/10/70	1/1000 1/10,000				
8/12/70	1/1000 1/10,000	<30,000 30,000	<1	<30,000 5,000	<1
8/14/70	1/1000	<3,000		<3,000	
8/18/70	1/1000 1/10,000	585,000 440,000	<1		
9/2/70	1/1000 1/10,000	<3,000 <30,000	<1	<3,000 <30,000	<1
9/29/70	1/1000 1/10,000	520,000 370,000	<1	13,000 20,000	<1
10/1/70	1/1000 1/10,000	<30,000 <30,000	<1	<30,000 <30,000	<1
10/5/70	1/1000 1/10,000	78,000 100,000	<1	21,000 30,000	<1
10/12/70	1/1000 1/10,000	<1,000 <10,000	<1	13,000 40,000	<1
10/28/70	1/1000 1/10,000	4,000 10,000	<1	<1,000 <10,000	<1
11/7&9/70 (Water)					

TABLE 13 Cont.

UF PERMEATE		RO FEED STAGE 1		RO FEED STAGE 2		RO FEED STAGE 3	
TPC	E.COLI	TPC	E.COLI	TPC	E.COLI	TPC	E.COLI
14,000	<1	4,000	<1	8,000	<1	4,000	<1
10,000		<10,000		<10,000		<10,000	
1,000	<1	4,000	<1	12,000	<1	3,000	<1
<10,000		20,000		16,000		<10,000	
67,000	3						
90,000							
<30,000	2	36,000	40	74,000	<1	140,000	<1
30,000		40,000		40,000		100,000	
780,000		650,000		650,000		650,000	
11,000	<1	17,000	12	46,000	63	156,000	208
10,000		30,000		20,000		160,000	
<3,000	<1	6,000	<1				
<30,000		<30,000					
190,000	<1	460,000	5	330,000	7	450,000	3
230,000		380,000		310,000		300,000	
140,000	<1	120,000	<1	160,000	<1	170,000	<1
180,000		160,000		190,000		140,000	
4,000	<1	17,000	<1	39,000	<1	57,000	<1
10,000		40,000		30,000		60,000	
21,000	6	220,000	<1	780,000	<1	650,000	<1
10,000		210,000		540,000		280,000	
41,000	<1	300,000	<1	520,000	<1	1,100,000	<1
50,000		340,000		460,000		1,600,000	
10,000	<1	<10,000	<1	<10,000	<1	<10,000	<1

TABLE 13 Cont.

LACTOSE CONCENTRATE		RO PERMEATE STAGE 1		RO PERMEATE STAGE 2		RO PERMEATE STAGE 3	
TPC	E.COLI	TPC	E.COLI	TPC	E.COLI	TPC	E.COLI
46,000 30,000	<1						
2,000 40,000	<1						
100,000 140,000	3	51,000 80,000	<1	91,000 130,000	<1	39,000 20,000	<1
160,000 110,000	<1	81,000 50,000	20	120,000 90,000	<1	110,000 70,000	<1
650,000		18,000		61,000		34,000	
143,000 70,000	>300	131,000 150,000	<1	440,000 340,000	<1	650,000 700,000	<1
<3,000 <30,000	<1	5,000 <30,000	<1	5,000 <30,000	<1	5,000 <30,000	<1
460,000 240,000	4	7,000 10,000	<1	1,000 <10,000	<1	16,000 10,000	<1
110,000 140,000	<1	150,000 240,000	<1	<30,000 <30,000	<1	<30,000 <30,000	<1
48,000 140,000	<1	20,000 20,000	<1	8,000 10,000	<1	62,000 60,000	<1
520,000 540,000	<1	20,000 10,000	<1	67,000 30,000	<1	520,000 460,000	<1
52,000 50,000	<1	92,000 70,000	<1	280,000 290,000	<1	980,000 1,400,000	<1
<10,000	0						

The tube ran only partially full, and microbiological growth was observed on the tube's inside surface. Proper cleaning and sanitizing of the tube was not possible.

Microorganism counts in RO permeates are based on samples taken from the module collection pans. These were generally not washed or sanitized, accounting for the relatively high levels of microorganisms on many dates. On days when the system was well sanitized (see for example, September 2), however, counts in the permeates were low.

Samples from all sampling points, except for the UF concentrate, showed contamination at some time. It is to be noted that proper cleaning and sanitizing was very effective in lowering these counts. Observe the data of October 5, which show very low counts, approaching levels for grade A milk. Similar results are seen for September 2.

The final entry in the table gives representative data for an experiment conducted over several days. At the end of the run on October 28, the system was cleaned and sanitized. At this time it was filled with water, which was sampled over a period exceeding one week. Bacteria counts for the system for November 7 and November 9 are shown, indicating the presence of very few residual microorganisms. Furthermore, when the system was stored until November 30, little bacterial growth occurred. This observation is based on the absence of off-odors and visible colonies.

It is concluded, therefore, that not only was the system design satisfactory from the point of view of sanitation, but also that the system could be stored for at least one month in water without substantial microbiological contamination.

SECTION VI

DEMONSTRATION PLANT DESIGN AND PROCESS ECONOMICS

PRELIMINARY DESIGN BASIS FOR AND DESCRIPTION OF 300,000 LBS/DAY PLANT

Design Basis for Ultrafiltration Section. The following design bases have been used for the UF section.

(a) 12 fold volumetric concentration.

(b) 20 hours operation per day in 2 batches; four hours for cleanup and sanitizing.

(c) Operating conditions:
- 20 gpm per tube (1 inch i.d.)
- 125°F
- 50 psi inlet and 15 psi outlet pressure to modules

(d) Average flux from pilot data of Table 11, corresponding to a pilot plant throughput of 1,000 lbs/hr, or 13.8 gfd.

(e) Membrane area requirements:

300,000 lbs/day:

$$\left(\frac{300{,}000}{8.5}\right) \times \left(\frac{24}{20}\right) \times \left(\frac{11/12}{13.8}\right) = 2810 \text{ ft}^2$$

$$\left(\frac{\text{lbs}}{\text{day}}\right)\left(\frac{\text{gals}}{\text{lbs}}\right)\left(\frac{\text{hours}}{\text{day}}\right) \times \left(\frac{\text{day}}{\text{operating hours}}\right) \times \left(\frac{\text{fractional } H_2O \text{ removal}}{\text{gal/day-ft}^2}\right)$$

Description of Ultrafiltration Section. A flow schematic for the ultrafiltration section is shown in Figure 53. The system contains 6 Abcor 480 ft^2 modules, providing a total membrane area of 2880 ft^2, slightly exceeding the required 2810 ft^2. The six modules are connected in a parallel flow arrangement. The overall flow design is analogous to that used in the pilot plant.

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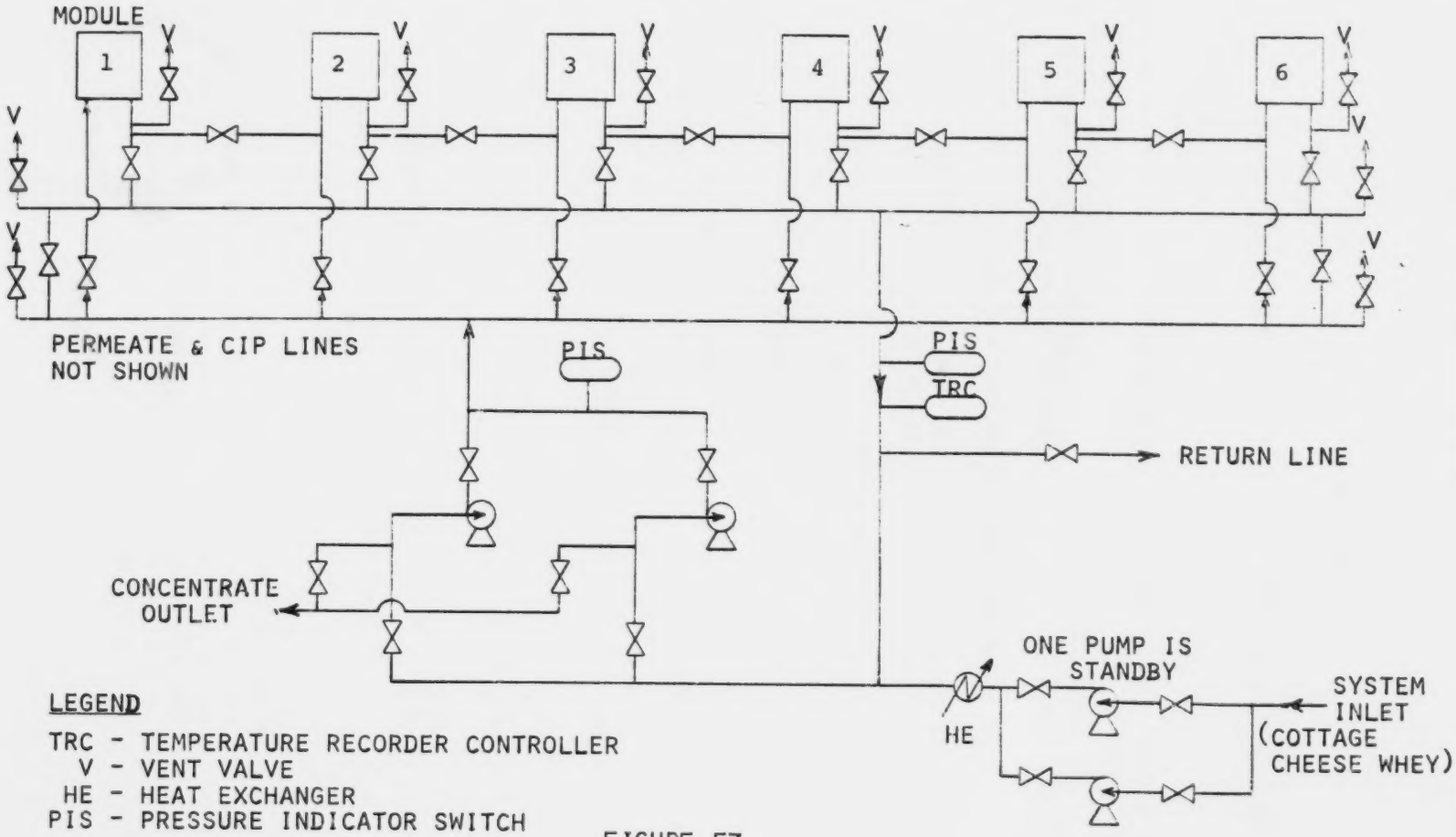

FIGURE 53

SIMPLIFIED FLOW DIAGRAM: ULTRAFILTRATION SYSTEM

Operation is on a batch basis. Whey is fed to a circulation loop from a storage reservoir. Whey from the loop is bled back to this reservoir, insuring mixing between the membrane loop and the storage reservoir. This maximizes both system capacity and protein yield. At the end of a batch operation, the protein concentrate is recovered from the system by flushing with water. Ultrafiltration permeate is withdrawn continuously and transferred to an interstage tank, prior to treatment by RO.

Raw whey is fed to the circulation loop with a 180 gpm feed pump. A second feed pump (spare) is included in the system on continuous standby. A high circulation rate through the modules is maintained by a 60 HP centrifugal pump. A second circulation pump (spare) is included in the system, also on continuous standby.

The ultrafiltration system contains a heat exchanger which allows for either heating or cooling of the raw whey. This permits control over operating temperature in the system. Additional instrumentation includes:

- high/low pressure shutdown switch
- conductivity guards on pumps
- a sight glass on each module to indicate flow
- a temperature recorder/probe, to measure, control, and record temperature
- pressure guages to measure inlet and outlet operating pressures (transmitted to control panel)
- process valves including two to each module (on/off valves), controlled from operating panel

Each module is fitted with CIP sprays and piping, and the system will include a 250 gpm spray pump, piping, and valves.

Total floor space to be occupied by the ultrafiltration unit will be approximately 1200 ft^2, in the form of a rectangle 30' x 40'.

Design Basis for Reverse Osmosis Section.

(a) Four-fold volumetric concentration of UF permeate to 21% solids.

(b) Twenty hours of operation per day on a continuous once-through basis. This is followed by a four hour cleanup and sanitizing.

(c) Operating conditions:
- $90^{o}F$
- 850 psi

(d) Average flux from pilot data of Figure 44 of 5.85 gfd.

(e) Membrane area requirements for 300,000 lbs of whey per day are:

$$\frac{(300,000) \times (11/12) \times (0.75)}{(8.5)} \times (\frac{24}{20}) \times (\frac{1}{5.85}) = 5100 \text{ ft}^2$$

Description of Reverse Osmosis Section. A flow schematic of the RO section is shown in Figure 54. The system has six parallel passes, each containing 63 Abcor RC 13-5 modules. These modules will have tubulence promoters and AS-197 membranes. Each parallel pass will contain 850 ft^2 membrane area, corresponding to a total plant area of 5100 ft^2. The design for each pass will be for once-through operation, with modules in a "Christmas-tree" arrangement. Several modules will be connected in parallel at the beginning of each pass, decreasing to a few modules at the end of each pass.

Each pass will have its own valving arrangement so that it can be shut down to replace any module which fails. This allows for membrane replacement without shutdown of the entire plant, although isolation of any single section of the plant will lead to a temporary reduction of capacity of 17%. Each pass will also have its source of filtered line water fed to the pass through check valves, permitting storage under line water pressure.

The membrane modules will be fed by a 25 HP positive displacement Quintuplex pump. A second pump will be included as a spare, in order to avoid plant shutdown in case of pump repairs.

Ultrafiltration permeate will be collected in a 6,000 gallon interstage tank, and will be pumped through the membrane modules on a once-through basis. Its temperature will be lowered from the UF operating temperature to that of the RO section by passage through a heat exchanger (HE). Cooling

water flow through the heat exchanger will be controlled by a temperature indicator/control device (TIC). The temperature indicator will also be connected to a recorder so that operating temperature can be monitored with time. Should the feed flow to the RO system be interrupted, a conductivity switch (CS) will shutdown the system. An accumulator after the high pressure pump will reduce pressure surges in the system to acceptable levels. A safety release valve (SRV) will vent to drain should the system be overpressurized.

Referring to Figure 54 a circulation loop will be required around the feed pump, and someflow will be circulated through a pressure let-down valve. Inlet and outlet pressures to the membrane modules will be measured. A low-pressure switch (PS) will shutdown the system in case of system failure. A back pressure regulator (BPR) on the concentrate line controls the system operating pressure, and the flow of the concentrate is measured with a flow indicator (FI).

By controlling the system downstream pressure with the back pressure regulator, and the recycle rate on the feed pump, the inlet pressure and the concentrate flow will be automatically fixed. If the amount of recycle on the feed pump is decreased, the inlet pressure will rise as will the lactose concentrate flow rate.

Permeate from the high pressure modules will be manifolded and discharged to the drain by gravity, or to a reservoir for reuse within the dairy.

Floor space requirement for the RO section will be approximately 750 ft^2, in the form of a 20' x 30' rectangle.

CAPITAL COSTS

Capital cost items are given in Table 14. Costs for the ultrafiltration and reverse osmosis sections (installed) are $217,000 and $360,000 respectively. Five tanks will be required. These include four 20,000 gallon tanks for raw whey and water storage. One 6,000 gallon surge tank will be needed for the UF permeate (feed tank for the RO section). Total costs for the tanks, including piping and valves, is projected to be $50,000.

Additional capital costs include $40,000 for installation, and $30,000 for a building to house the plant. Total capital costs for the plant are projected to be $697,000.

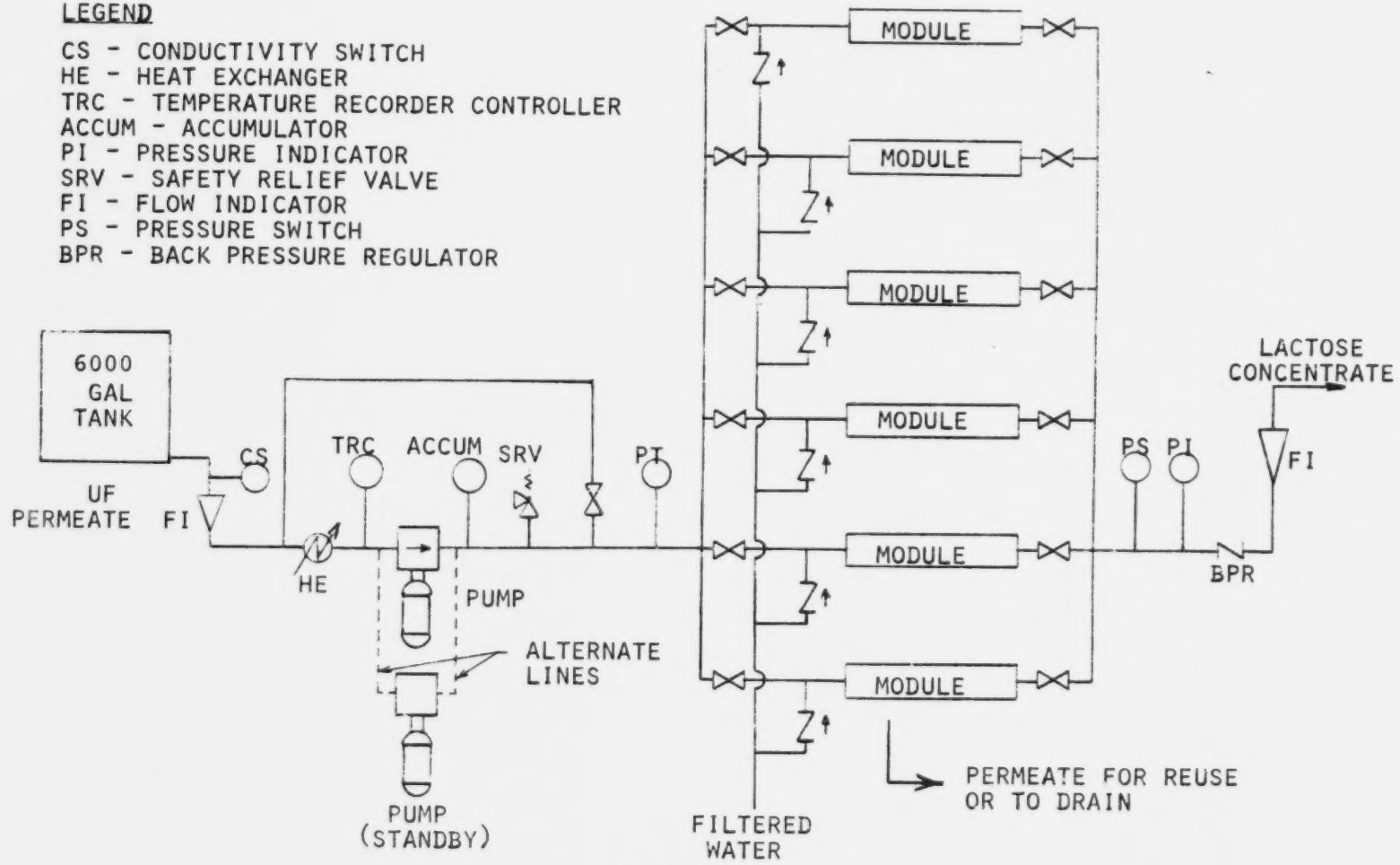

FIGURE 54

SIMPLIFIED FLOW DIAGRAM: REVERSE OSMOSIS SYSTEM

TABLE 14

PROJECTED CAPITAL COSTS FOR 300,000 LBS. WHEY/DAY PLANT:

Equipment	
Ultrafiltration Section (CPI tanks included), installed	$217,000
Reverse Osmosis Section, installed	360,000
Tanks	
4 - 20,000 gals. plastic silos for whey and water storage	
1 - 6,000 gals. UF permeate surge tank	
Total for 5 tanks with piping and valves	50,000
	627,000
Installation Costs (other than membrane equipment)	40,000
Building Costs (3000 ft^2)	30,000
TOTAL CAPITAL COSTS	$697,000

OPERATING COSTS

Operating costs are shown in Table 15. Among the items listed, costs for power and cooling water are based on actual operating data from the pilot plant. The membrane equipment itself, has been depreciated on two bases. Membrane modules, less the annual charge for membrane replacement, have been depreciated over five years. The associated membrane hardware (pumps, valves, controls, etc.) has been depreciated over ten years, as have been the remaining capital cost items.

The annual operating costs will be approximately $220,000.

PROJECTED BY-PRODUCT VALUES AND PROCESS PROFITABILITY

Fractions obtained from the membrane process have useable characteristics. In treating 96,000,000 lbs of cottage cheese whey per year, 576,000 lbs of protein (at 0.6% in whey) and 4,320,000 lbs of lactose (at 4.5% in whey) will be recovered. The value for these by-products when used as food ingredients will be substantial in dollars. The net annual profit, the difference between income and operating expenses, is expected to show a highly favorable return on investment.

COSTS FOR OTHER COMMERCIAL PLANTS

It should be noted that the capital costs projected here are for the first combined UF and RO plant designed specifically for use in a dairy. The costs of subsequent plants may be significantly lower. Operating costs would also be expected to be reduced. The construction and operation of the full-scale plant in Phase II of the project will provide more accurate cost information required for widespread acceptance of UF and RO.

TABLE 15

PROJECTED OPERATING COSTS FOR 300,000 LBS WHEY/DAY PLANT

Basis: 320 days/yr or 7680 hrs/yr	
Labor and Overhead:	
Supervisory: 3 hrs/day x 320 days/yr @ $5.00/hr	$ 4,800
General Utility: 7,680 hrs x $3.00/hr	23,040
Lab Tester: 2 hrs/day x 320 days/yr @ $3.00/hr	1,920
Mechanic: 1 hr/day x 320 days/yr @ $4.00/hr	1,280
	$ 31,040
Fringe and overhead @ 40% of wages	12,400
Total Labor	$ 43,440
Annual Membrane Replacement Cost @ $5/ft^2	39,900
*Power @ 1.2¢/kwh	5,000
*Cooling Water	4,000
Steam	3,000
Cleaning Chemicals ($10/day)	3,200
Disposables (e.g., pump gaskets, seals etc)	3,200
Professional Services (Outside maintenance costs, engineering services, consultants)	10,000
Interest and Taxes @ 3% of Capital	20,910
Depreciation:	
Membrane Modules ex membranes @ 20%	42,700
Membrane Process Hardware (pumps, valves, controls, etc.) @ 10%	32,400
Remaining Capital Items @ 10%	12,000
Projected Annual Operating Costs	$219,750

*Actual costs based on scale-up of pilot plant utility usage.

SECTION VII

INDUSTRY INTEREST (INCLUDING PUBLICATIONS)

The Crowley's demonstration project will be a success for EPA only if cottage (and other) cheese makers throughout the country accept ultrafiltration and reverse osmosis as whey treatment processes in their own plants.

Measures of industry acceptance are the papers and publications reporting on the project (Table 16) and the number of visitors to the pilot plant facility (Table 17).

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TABLE 16

PUBLICATIONS AND PAPERS

1. R.R. Zall, talk given at Metropolitan Dairy Technology Society, New York, N.Y., (March, 1970).

2. B.S. Horton, et. al., "Membrane Separation Processes for the Abatement of Pollution from Cottage Cheese Whey", presented at the Cottage Cheese and Cultured Milk Products Symposium, University of Maryland (March 11, 1970).

3. B.S. Horton, "Prevents Whey Pollution-Recovers Profitable Byproducts", Food Engineering, Vol. 42, No. 7, pp. 81-83 (July, 1970).

4. B.S. Horton, et. al., "New Method for Economical Control of Pollution Caused by Cheese Wheys", presented at SOS/70, Washington, D.C., (August 14, 1970).

5. R.L. Goldsmith, et. al., "Recovery of Cheese Whey Proteins through Ultrafiltration", presented at SOS/70 Washington, D.C. (August 14, 1970).

6. R.R. Zall, talk given at American Cultured Diary Products Institute, Cornell University, Ithaca, N.Y. (September, 1970).

7. B.S. Horton, "Ultrafiltration and Reverse Osmosis for Processing Cottage Cheese Whey", presented at American Cultured Dairy Products Institute Annual Meeting, Cornell University, Ithaca, New York (September 9, 1970).

8. B.S. Horton, "Ultrafiltration and Reverse Osmosis for Processing Cheese Wheys", presented at South Dakota State Dairy Association Annual Convention, Sioux Falls, S. Dakota (September 22, 1970).

9. B.S. Horton, et. al., "Membrane Separation Processes for the Abatement of Pollution from Whey", XVIII International Dairy Congress, Sydney, Australia (October 12-16, 1970).

10. B.S. Horton, talk given for the Massachusetts Institute of Dairy Science, Boston, Mass. (November, 1970).

11. Goldsmith, R.L. et. al., "Industrial Ultrafiltration", presented at American Chemical Society Symposium on Membrane Processes, Chicago, Illinois, (September 13-18, 1970).

12. Goldsmith, R.L., et. al., "Membrane Processing of Cottage Cheese Whey for Pollution Abatement", presented at Second National Symposium on Food Processing Wastes, Denver, Colorado, (March 23-26, 1971).

13. Horton, B.S., talk given at Quality Chek'd Products Annual Production Meeting, Memphis, Tennessee, (March, 1971).

14. Goldsmith, R.L., et.al., "Industrial Ultrafiltration" in Membrane Processes in Industry and Biomedicine, Plenum Press, 1971, Pages 267-300.

15. Horton, B.S., et.al., "Membrane Separation Processes for Treatment of Whey and Rinse Waters in Cottage Cheese Plants of All Sizes", 1971 Conference of American Cultured Dairy Products Institute, Athens, Georgia, (September 8-9, 1971).

16. Horton, B.S., talk given at Nordica Foods Annual Seminar, Sioux Falls, South Dakota, (October, 1971).

17. Zall, R.R., et.al., "Membrane Processing of 300,000 lbs/day Cottage Cheese Whey for Pollution Abatement, Phase II", presented at AIChE 64th Annual Meeting, San Francisco, California (November 28-December 2, 1971).

18. Horton, B.S., et.al., "Whey Processing with Sanitary, CIP Membrane Equipment", presented at 1971 Winter Meeting, ASAE, Chicago, Illinois, (December 7-10, 1971).

TABLE 17

VISITORS TO WHEY PILOT PLANT AT CROWLEY'S MILK CO.

NAME & TITLE	COMPANY & ADDRESS	DATE VISITED
T.W. Brooks, Ph.D. Section Leader	Calgon Corp. Calgon Center P.O. Box 1346 Pittsburgh, Pa. 15230	10/5/70
Professor James Harper	Ohio State University Columbus, Ohio	10/14/70
Mr. J.L. Maubois Mr. J.P. Barbier	Institut National de la Recherche Agronomique Laboratoire de Recherches de Technologie Laitiere 65, Rue de Saint-Brieuc 35 Rennes, France	10/20/70
James Garrison, V.P. Mfg. Delbert Harmon Dr. Larry Claypool V.P. Research Dick Bonney Mgr. Mfg. Operations Burdet Heinemann, Pres.	Mid-America Dairymen, Inc. P.O. Box 1837 S.S. Station Springfield, Mo. 65805	10/19/70
Dr. Curtis Hallstrom Charles Hungerford	General Mills Central Research Lab. James Ford Bell Res. Center 9000 Plymouth Avenue Minneapolis, Minn. 55427	10/19/70
Russell W. Carnahan	Kraftco Corp. Research and Dev. Div. 801 Waukegan Road Glenview, Ill. 60025	10/27/70
Marty Engel	Kraftco Corp. (see above)	10/28/70
Eugene Kolen	"	"
Hubert Patta Director	Cooperative du Val d'Or 45, Saint-Aignan-des-Gues Loire-et-Ch. France	10/28/70

NAME & TITLE	COMPANY & ADDRESS	DATE VISITED
L. Benghouzi Ingenieur	Direction Departmentale de l'Agriculture du Loiret 21, rue Eugene-Vignat 45 Orleans, France	10/28/70
Lloyd J. Peterman	A&P National Dairy Div. 102 Revere Drive Manitowoc, Wisc. 54220	9/8/70
Edward S.K. Chian, Sc.D. Mgr. of Technology	American Standard Conseps P.O. Box 5000 Hightstown, N.J. 08520 (Abcor)	8/21/70
William O. Miller	Coca Cola (see below)	8/19/70
Anton Amon	Coca Cola Export Corp. 515 Madison Avenue New York, N.Y. 10022	8/19/70
K.J. Kirkpatrick	New Zealand Dairy Research Institute P.O. Box 1204 Palmerston North, N.Z.	9/19/70
Avelino Salgado de Oliveira, Jr. Engineer	Lacticinios bom Pastor, LDA Rua Carlos Mardel 38-C Lisboa-1 Portugal	
Victor Moreno, Ph.D.	General Foods Tarrytown, N.Y.	9/29/70
R.E. Farrar Exec. V.P.	Dairy Research Inc. 120 Eastman Building Arlington Heights, Ill. 60004	9/30/70
Steven B. Chall Sr. Engineer	General Foods Technical Center Tarrytown, N.Y. 10519	9/29/70
Arno Huste Sr. Research Specialist	General Foods (see above)	9/29/70
George Rey	E.P.A. Alexandria, Va.	9/17/70

NAME & TITLE	COMPANY & ADDRESS	DATE VISITED
Bob Burm	E.P.A.	9/23/70
E.T. Wilhelm Manager	Central Soya Process & Product Dvlmt. 1825 N. Laramie Avenue Chicago, Ill.	10/7/70
Charles Zanzig	Foremost Foods Co., R&D 6363 Clark Avenue Dublin, California 94566	10/10/70
Dave Hennigh Dept. Manager	Safeway Stores 2538 Telegraph Avenue Oakland, California 94612	11/9/70
Professor Richard March	Cornell University Ithaca, New York	10/30/70
George Walgrove, Plant Sup. Walter Kneeland Proj. Leader	H.P. Hood & Sons 500 Rutherford Avenue Boston, Mass. 02129 Plant at St. Albans, Vt.	11/10/70
Gerald W. Smith Gen. Manager John B. Townsend	Meyer-Blanke Co. 5432 Highland Drive St. Louis, Missouri 63110	11/30/70
H.N. Jensen Assistant Director	Dart Industries P.O. Box 3157 Terminal Annex Los Angeles, California 90054	11/23/70
Stephen J. Wolff	Pevely Milk Company 1001 South Grand Blvd. St. Louis, Missouri 63104	11/30/70
Allyn Richardson	E.P.A. N.E. Region JFK Federal Building Government Center Boston, Mass. 02203	11/30/70
A. de la Bourdonnaye Ingenieur	Ministere de l'Agriculture Dir. des Industries Agricoles et Alimentaires 3, Rue Barbet-de Jouy Paris VIIe France	11/70

SECTION IX

ACKNOWLEDGEMENTS

The support of the project by the Environmental Protection Agency and the help provided by Mr. William Lacy, Mr. George Keeler, and Mr. Allyn Richardson, the Federal Grant Project Officer, is acknowledged with sincere thanks. The project directorship was under the responsibility of Robert R. Zall of Crowley's Milk Co.

Dr. Robert L. Goldsmith of Abcor, Inc. held program responsibility for Abcor, Inc's. subcontracted activities, and prepared this report with the aid of Mr. Bernard S. Horton (Abcor). Messrs. Sohrab Hossain (Abcor) and Michael Tan (Abcor) executed the prototype experimental program. Dr. David J. Goldstein (Abcor) was in large part responsible for the design and construction of the pilot plant. Many helpful discussions were held with Drs. Robert S. Timmins and William Eykamp (Abcor), and Professors Steven R. Tannenbaum and Tony Sinskey (M.I.T., Dept. of Nutrition and Food Science).

Mr. Patrick Crowley at Crowley's Milk Co., aided in the operation of the pilot plant. Much of the analytical work at Crowley's Milk Co. was performed by Mrs. Anna Wichtowska, Research Associate. Substantial assistance and support for the project were provided by Mr. William Burtis of Crowley's, the project finance officer, and also Messrs. Vincent and Elmer Crowley.

Membrane equipment formerly fabricated by the ConSeps Department of American Standard Corp. is presently manufactured by Abcor, Inc. All membrane equipment for the Phase II, 300,000 lbs/day plant will be manufactured by Abcor, Inc.

SECTION X

REFERENCES

1. Anonymous, Volume III, Industrial Waste Profiles, No. 9-Dairies, "The Cost of Clean Water", FWPCA, Washington, D.C., September, 1967.

2. McDonough, F.E., "Whey Concentration by Reverse Osmosis", Food Engineering, Vol. 40, No. 3, March, 1968.

3. Marshall, P.G., Dunkley, W.L., Lower, E., "Fractionation and Concentration of Whey by Reverse Osmosis", Food Technology, Vol. 22, No. 8, pp. 37-44, August, 1968.

4. Anonymous, "USDA Studies Reverse Osmosis as Whey Disposal Method", New Release No. USDA 1396-68, Washington, D.C., May 1, 1968.

5. deFilippi, R.P., Goldsmith, R.L., "Application and Theory of Membrane Processes for Biological and Other Macromolecular Solutions", Membrane Science and Technology, ed., James E. Flinn, Plenum Press, 1969.

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1 Accession Number	2 Subject Field & Group	SELECTED WATER RESOURCES ABSTRACTS INPUT TRANSACTION FORM
W	05D	

5 Organization

Crowley's Milk Company
Binghamton, New York

6 Title

Membrane Processing of Cottage Cheese Whey for Pollution Abatement

10 Author(s)

Goldsmith, R.L.
Horton, B.S.

of Abcor, Inc.
Cambridge, Mass.

16 Project Designation

EPA, WQO Project 12060 DXF

21 Note

22 Citation

23 Descriptors (Starred First)

*Cottage Cheese Whey Treatment, Cottage Cheese Processing, Whey, Food Processing, Membrane Separation, Pollution Abatement, Reverse Osmosis, Ultrafiltration

25 Identifiers (Starred First)

*Cottage Cheese Whey, *Whey, Whey Treatment, Dairy Waste, Waste Treatment, Membrane Processes

27 Abstract

A two-step membrane process was demonstrated for treatment of cottage cheese whey. The process produces valuable protein and lactose by-products while reducing BOD of effluent whey. The process was studied in detailed prototype experiments at Abcor, Inc., Cambridge, Mass., and a 10,000 lbs/day pilot plant at Crowley's Milk Company, Binghamton, New York.

In the two-step process, a protein concentrate is first recovered by ultrafiltration. In the second step, ultrafiltration permeate (de-proteinized whey) is concentrated by reverse osmosis providing a lactose concentrate. The protein concentrate can be further concentrated and/or dried; the lactose concentrate can be further concentrated and lactose recovered by crystallization, or otherwise processed. Alternatively, the membrane concentrates are usable as fluid products.

Operation of the pilot plant was successful and almost troublefree. BOD reduction of raw whey was about 97%, from initial values of about 35,000 to less than 1,000 mg/l. Membrane life was excellent, and membrane fluxes were economically high.

The pilot plant produced protein and lactose products with low total plate and nil coliform counts. Using the cleaning procedure developed in prototype tests, total plate counts were typically below 50,000 org/ml.

Process economics are presented.

Abstractor: Robert Goldsmith

Institution: Abcor, Inc., Cambridge, Massachusetts

WR:102 (REV. JULY 1969)
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